Historic, archived document

Do not assume content reflects current scientific knowledge, policies, or practices.

U. S. DEPARTMENT OF AGRICULTURE.
DIVISION OF CHEMISTRY.
BULLETIN No. 24.

PROCEEDINGS

OF THE

SIXTH ANNUAL CONVENTION

OF THE

ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS,

HELD AT

THE U. S. DEPARTMENT OF AGRICULTURE
SEPTEMBER 10, 11, AND 12, 1889.

METHODS OF ANALYSIS OF COMMERCIAL FERTILIZERS, CATTLE FOODS, DAIRY PRODUCTS, AND FERMENTED LIQUORS.

EDITED BY

HARVEY W. WILEY,
SECRETARY OF THE ASSOCIATION.

PUBLISHED BY AUTHORITY OF THE SECRETARY OF AGRICULTURE.

WASHINGTON:
GOVERNMENT PRINTING OFFICE.
1890.

PREFATORY NOTE.

SIR: I have the honor to submit herewith, for your inspection and approval, the report of the proceedings of the sixth annual meeting of the Association of Official Agricultural Chemists. The importance of the work of this association is now recognized by the agricultural chemists not only of this country, but also of Europe. The progress in methods and accuracy of analysis made since the organization of this association has rendered the work of agricultural analysis more uniform and authoritative, and this has been so radical that results obtained by other methods are regarded with suspicion if not with disfavor. While this is true it is not claimed that our methods are perfect. It is realized that new investigations and new discoveries will continue to make changes in methods not only desirable but necessary. For this reason the wisdom of annual conventions can not be doubted. The aid extended heretofore by the United States Department of Agriculture has greatly helped to the accomplishment of our purposes, and in behalf of the association, and as the editor of its proceedings, I ask its continuance.

Respectfully,

H. W. WILEY,
Chemist.

Hon. J. M. RUSK,
Secretary.

PROCEEDINGS OF THE SIXTH ANNUAL CONVENTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, HELD AT WASHINGTON, SEPTEMBER 10, 11, AND 12, 1889.

FIRST DAY.

MORNING SESSION TUESDAY.

In accordance with the call of the executive committee, the association met in the Seed Division of the Department of Agriculture at 9:30 o'clock, the president, Mr. John A. Myers, in the chair.

The following members and "others interested in the objects of the Association" were present:

Hon. Edwin Willits, Assistant Secretary of Agriculture.

The president, Mr. John A. Myers, director of the West Virginia Agricultural Experiment Station, Morgantown, W. Va.

The vice-president, Mr. M. A. Scovell, director of the Kentucky Agricultural Experiment Station, Lexington, Ky.

The secretary, Mr. Clifford Richardson, chemist to the District of Columbia.

Of the executive committee, Messrs. H. W. Wiley, of Washington, D. C., and William Frear, of State College, Pa., and the following members and participants:

Messrs. James T. Anderson, of Auburn, Ala.; W. O. Atwater, of Washington, D. C.; H. B. Battle, of Raleigh, N. C.; Walker Bowman, of Blacksburgh, Va.; W. B. Burney, of Columbia, S. C.; G. C. Caldwell, of Ithaca, N. Y.; F. W. Clarke, of Washington, D. C.; F. E. Engelhardt, of Syracuse, N. Y.; G. S. Fellows, of Washington, D. C.; Richard H. Gaines, of Richmond, Va.; W. J. Gascoyne, of Baltimore, Md.; H. H. Harrington, of College Station, Tex.; J. D. Hird, of College, Md.; George L. Holter, of State College, Pa.; W. L. Hutchinson, of Agricultural College, Miss.; E. H. Jenkins, of New Haven, Conn.; R. C. Kedzie, of Agricultural College, Mich.; B. W. Kilgore, of Agricultural College, Miss.; N. T. Lupton, of Auburn, Ala.; J. B. McBryde, of Columbia, S. C.; H. B. McDonnell, of State College, Pa.; Fred. W. Morse, of Hanover, N. H.; H. H. Nicholson, of Lincoln, Nebr.; H. J. Patterson, of College, Md.; Edgar Richards, of Washington, D. C.; Norman Robinson, of Lake

City, Fla.; D. B. Ross, of Baton Rouge, La.; C. Morton Strahan, of Athens, Ga.; Bruno Terne, of Philadelphia, Pa.; E. B. Voorhees, of New Brunswick, N. J.; Robert B. Warder, of Washington, D. C.

The president called the convention to order and introduced Hon. Edwin Willits, Assistant Secretary of Agriculture, who spoke as follows:

ADDRESS OF HON. EDWIN WILLITS.

Mr. President and gentlemen of Association of Agricultural Chemists: It gives me great pleasure to welcome you to the Department of Agriculture, and to express the gratification we feel in the honor of your presence. Since you last met Congress has added to the dignity of this Department by making it an executive branch of the Government, and in so doing has acted in accord with the new impulse in favor of scientific agriculture. I speak of it as a new impulse. I ought rather to have said *added* impulse, for the movement towards higher work and more thorough investigation is not new, but from small beginnings has gone on growing and increasing with the running years. From one end of the country to the other, I may say from all points of the civilized globe, has come the demand that agriculture shall be recognized, not simply as the prime element in national prosperity, but as occupying the platform for legitimate affirmative legislation, as much as national defense, foreign and internal commerce, or international relations. This feeling found recent expression in the establishing of experiment stations in all the States under the financial endowment of the national Government, and was followed as a logical necessity by an enlargement of the scope and standing of this Department. The work committed to the stations is recognized as national, and though local in its application the co-operation of this Department with the stations emphasizes its nationality. It adds to our pleasure, therefore, in having you with us to-day, that this Department is a co-worker with you in the wide field which you represent, and for this reason can more fully appreciate your wants and work.

Under the act of March 2, 1887, it is the duty of this Department—

> to furnish forms, as far as practicable, for the tabulation of results of investigations and experiments, and to indicate from time to time such lines of inquiry as shall seem most important; and in general to furnish such advice and assistance as will best promote the purposes of this act.

Under act of March 2, 1889, the Department is given $15,000—

> to enable the Secretary of Agriculture to compare, edit, and publish such of the results of the experiments made by the stations as he may deem necessary.

It will be seen that the relations of this Department with the stations are intimate and important; that while the power to control their work is not conferred, the right to suggest, to indicate lines of work, to furnish advice and assistance, is clear and positive; and the duty to scrutinize and sift your work for publication is unquestioned. It is, therefore, not only appropriate, but in a sense necessary, that there should be a fair understanding between us, not only of what is to be done, but of the methods adopted and the forms used. Most of the original work is to be done by the stations, but the leading thought in the legislation seems to be that the lines of work should center in the Department, though not emanate from it, except in the suggestions and advice that would be incident to an agent competent to compare and edit the results. To be better competent to furnish advice and assistance, we wish to be associated with you, to confer with you, and to determine how we best can aid you in promoting the purposes of the act of March 2, 1887.

But this is not all. The work in chemistry is not limited to experiments and investigations in agriculture. Chemistry is broader than even that broad field. You meet here as chemists, working most of you in a special line, but made more competent for that work by not being limited to it. The best specialist is the man who is in touch with all lines. It is the broad man that is the most forceful when his energies are concentrated on a single point. So your deliberations here must have as final arbiter those general principles without which none of your processes will be a permanent success. Therefore, it is to be expected that what you do, or conclude to do, what you report as having done and having discovered will be based upon chemistry and not upon charlatanism. For this reason the Department of Agriculture esteems that as in the end the most practical which has at its back the certification of science, and it prides itself in calling to its aid the best that science can confer; and in its Chemical Division it hopes you will find workers worthy of co operating with you as associates in your deliberations.

A few words on the importance of your work, and I am done. Since the first experiment station was established to the present, there has been great diversity of opinion as to what shall be the scope of its work, but from the time of Liebig down there has, so far as I know, never been a suggestion of one without chemistry. In fact, in them all chemistry has been the leading factor, and while more recently they have widened the scope, and justly so, of their work, the final test is still largely found in the crucible. There is perhaps a philosophical reason for this. Experiments and investigations generally adopt one of three lines: (1) constructive, (2) preventive, and (3) development by selection and cultivation. The last aids nature, pruning her exhuberance, selecting her strong points, and clearing the way for higher development. The second discovers and kills the noxious parasite that preys upon vitality, so that nature shall not be abortive. The first is more fundamental. In a sense it goes to the bottom of things. It perhaps answers more whys and suggests more wherefores than any other science. In its analysis it gets nearer the elemental forces, and handling them singly can combine them more readily and rationally. Chemistry, analytical in its inception, is essentially constructive in its results. It first finds the defect and then supplies the want. It puts iron into the blood. At least that is the way it looks to an outsider; and the generations that looked on with awe while the "caldron cooked the hell-broth," have left as a legacy the superstition that an art or a science that can take nature to pieces, as it were, must not be ignored in the make-up of an experiment station whose prime purpose shall be to steal from nature some of her secrets as trophies in scientific progress. So it is no wonder that everybody concedes that chemistry shall be consulted in our investigations. It can not be denied, however, that other sciences are contesting the field, and I would not be loyal to the other divisions of the Department, of which I am the nominal head, if I did not claim for them that they are entitled to equal consideration, and that for practical results they are, some of them at least, showing themselves worthy of it.

Gentlemen, you have history at your back, and you will be degenerate sons of illustrious sires if you abandon a single intrenchment, or abate in any measure your estimate of the importance of your work. But your deeds must justify your claims, and the better to equip yourselves for the contest you must take advantage of every means of information, and there is no better means, among many, all to be adopted, than conferring together over this field to be traversed and won, over the implements to be used, and over the processes employed. May you have a profitable session. Again thanking you for your presence here, and welcoming you within walls rather limited, and in a sense inhospitable, but hoping that the Congress about to convene shall consider our growing wants and shall give us laboratories and lecture-rooms that shall more fully express the hospitality of our hearts, I abandon the platform to the purposes for which you are assembled.

In response to the welcome of Secretary Willits, the president addressed the convention as follows:

ADDRESS OF MR. JOHN A. MYERS, PRESIDENT.

Gentlemen of the Association of Official Agricultural Chemists, it is by the kind invitation of the Assistant Secretary of Agriculture, extended to this association in the following communication, that this annual meeting was called by the executive committee to assemble in these halls under the auspices of the Department of Agriculture:

UNITED STATES DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
Washington, D. C., June 26, 1886.

DEAR SIR: I desire through you to extend an invitation to the Association of Official Agricultural Chemists to hold its next meeting in the buildings of the Department of Agriculture at such time as the executive committee may deem best.

I beg to assure you of my hearty sympathy with the objects of this association, and of my appreciation of the good work which it is doing for the benefit of agricultural science in this country. I will promise to co-operate with you in every possible way to secure a successful meeting, and to place before the agricultural chemists of the country the proceedings of the convention in such form as may be most convenient.

Respectfully,

EDWIN WILLITS,
Assistant Secretary.

Prof. J. A. MYERS,
President Association of Official Agricultural Chemists,
Morgantown, W. Va.

MORGANTOWN, W. VA., *July* 1, 1889.

MY DEAR SIR: Permit me upon the part of the Association of Official Agricultural Chemists to accept your kind invitation to hold the next meeting of this association in the buildings of the Department of Agriculture. In accepting, allow me to assure you that the association appreciates very highly your interest in the work in which it is engaged, and the honor that you confer upon it in extending to it this hearty invitation. The official agricultural chemists certainly have many reasons to be thankful to the presiding officers of the Department of Agriculture for the kindnesses extended to the association in the past, and it is especially gratifying to us to know that our scientific work is to receive the sympathy and encouragement of the present administration. I know that I express the sentiments of every member of the association when I convey to you our hearty thanks for your kind offer, and trust that the meetings may prove of sufficient value to have the continued recognition of the Government.

I have the honor to be, sir, yours very truly,

JOHN A. MYERS,
President of Association of Official Agricultural Chemists.

Hon. EDWIN WILLITS,
Assistant Secretary of Agriculture, Washington, D. C.

The distinguished Assistant Secretary of Agriculture has addressed a personal invitation to every official agricultural chemist in this country and to all of the agricultural experiment stations, inviting them to be represented at this meeting and to participate in its deliberations. There is always a standing invitation to the analytical chemists of the country to be present and to enter heartily into its discussions.

Our proceedings for several years have been published as a bulletin by the Division of Chemistry, and to the distinguished chief of this division we are indebted for many courtesies.

Recently another warm friend of the association has arisen in the Department in the form of the chief of the Office of Experiment Stations, who has kindly sent an invitation to all of the agricultural experiment stations of the country to be present at this meeting. So much interest in this organization manifested by the honorable Secretary of Agriculture and his staff makes us feel doubly welcome in coming together today in the halls of this Department to consider the best means of correcting, improving, and extending the methods of chemical analysis in this country.

Among our ranks I see men who have traveled farther than from St. Petersburg to Rome, or from London to Constantinople, to meet their brother chemists and to discuss for a few days the principles upon which rest the success or failure of their work. These men are not hampered by any political sentiment nor constrained in their action by any private or personal object, but are seeking the truth. This organization, in some respects, has nothing to compare with it in the world. It was demanded by the commercial interests of the country, and was necessary as a means of self-protection upon the part of the chemists from the onslaughts made upon them by parties exposed and brought into court on account of frauds detected.

It is aiming to lay a foundation so solid that every court in this land must respect its conclusions, and every analytical chemist, whether he lives in this country or elsewhere, must be forced either to practice or admit the advantages and correctness of our system of analyses. Step by step we have advanced through the difficult problems before us, feeling that we have steadily gained ground and that we are molding public sentiment in a manner that must redound to the advantage of the whole country and secure the highest respect for the science to which we are devoting our lives. We have in five years developed under the pressure to which we have been subjected an association which is recognized by one of the most progressive governments of the world as being reliable; its conclusions are very largely accepted in the courts of the country and adopted as trustworthy by a number of our State legislatures.

Our constitution states that the first object of this association shall be "to secure uniformity and accuracy of methods, results, and modes of statements of analysis of fertilizers, soils, cattle food, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for a discussion of matters of interest to agricultural chemists." The importance of securing accuracy of method and uniformity of statement of analyses of fertilizers was really the prime element leading to the formation of this organization. So much confusion had existed both in methods of analyses and statements, out of which grew in many cases serious litigation, the chemists being dragged into the courts as witnesses, abused by the attorneys upon one side and praised for their results by those upon the other side, that it became necessary to protect not only the chemists themselves, but the reliability of their science by some system of organization and mutual understanding.

Early in the history of its organization an effort was made by some injudicious and perhaps inexperienced parties to get control and force upon the official chemists of the country analytical methods which experience had demonstrated unreliable. This led to certain limitations in the voting power of parties attending the convention being incorporated into our constitution, and hence we find an article to this effect: "Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers." But the association has always extended an invitation to all analytical chemists in the country engaged in any kind of analytical work bearing directly or indirectly upon agriculture. They have been invited to be present and to participate in the discussions, to give this association the benefit of their experience and advice, and to help us arrive at methods of analysis which will answer all of the demands of modern times. We strive, first, to secure accuracy; second, rapidity and adaptability of methods of analysis; and, thirdly, uniformity in stating results.

The good effect of our work may be seen in the influence that it has exerted upon the fertilizer trade of the country. There is now, I believe, no State in this country having a fertilizer control or inspection that does not recognize by special enactment the methods of analysis of fertilizers adopted by it. This matter alone affects commercial transactions in this country involving millions of dollars. In many of our cities laws have been passed requiring regular and constant inspection of milk, butter, cheese, and other agricultural products offered for sale, and Congress has passed certain acts regulating the sale of artificial dairy products. There is also a growing demand throughout the country requiring that liquors, both fermented and distilled, shall receive the chemist's certificate before being offered for sale. If this association continues to maintain its proper attitude and high sense of the responsibilities resting upon it, we may safely say that the time is not far distant when its deliberations will be considered of national importance, and its conclusions will virtually become the law regulating the sale of nearly every agricultural and food product offered in our cities.

I have been glancing over such statistics as were accessible to me to gain if possible some idea of the influence that this association exerts and is likely to exert upon the commercial activity of our country. In 1885 the sugar and molasses brought into this country amounted in money value to $86,369,414. In addition to this, other agricultural products which are subjected to more or less rigid chemical examinations and inspections were imported to the value of nearly $100,000,000. The fertilizers and heavy chemicals imported from South American countries alone amounted to about $2,000,000. If to this we add a long list of less important products imported in quantities ranging in value from a few thousands up to several millions of dollars, we see that this association has met to discuss methods of analysis effecting the valuations of importations approaching $200,000,000 in value. When we add to this the enormous amounts involved in home products, we have a sum so great that I scarcely dare to name it. Suffice it to say that it represents more than the entire banking capital of the country.

I grant freely that every shipment of fertilizers, every cargo of sugar, and every gallon of milk or pound of butter may not be inspected; but the Government is maintaining and is likely to increase its facilities for extending these inspections. Public sentiment in favor of them is constantly increasing, and this association if it maintains the proper conservative attitude and strict adherence to scientific principles observed in the past, must in the near future virtually dictate the laws regulating all chemical inspections in this country.

Time does not permit me to go into the minutia of this statement, but I believe that a careful investigation will develop the fact that there is no other association of scientific men in the world that should be more careful in its deliberations and more cautious in the conclusions arrived at than this one. The official analytical chemist occupies a peculiar position. He is the arbiter to whom must be referred questions involving the profit and loss upon an enormous amount of capital, besides those involving the enforcement of law, the punishment of violators of the law, and the protection of the innocent.

The value of the work undertaken by this association is so apparent that its system of investigation is being adopted by other associations in regard to the applications of chemistry to various branches of industry. Last year the British Association for the Advancement of Science, Section B, appointed a committee to consider the best methods of establishing international standards for the analyses of iron and steel. In this it was proposed that an effort should be made to secure uniformity of methods of analyses and of statements in the United Kingdom, America, France, Germany, and Sweden. Uniform samples were to be sent to chemists of recognized repute in this kind of work in the several countries for analysis. The mean of the results of the analyses attained in this way was to be considered as representing the composition of the standards. The number of samples of steel were limited to four, containing as nearly as possible 1.30 per cent., .80 per cent., .40 per cent., .15 per cent., total carbon, respectively. In addition to this the determination of the amount of carbon present in each condition; phosphorus, sulphur, silicon, manganese, and chromium also to be determined. These were to be so divided that other chemists in the respective countries might have samples of these standards for comparison. It is proposed in this way to establish a uniform system of comparing the chemical qualities of iron and steel in the respective countries. So far as I am aware, no results have as yet been arrived at by this committee, but it is presumed that they are working upon them.

The Germans have several associations similar to this one, none of which, however, have undertaken as extensive a line of comparative work as ours. England has its society of analysts, which does not appear to have any regular system of comparing the work of its members, each analyst apparently standing upon his own responsibility and accepting or rejecting the usual processes of analyses as may seem to him advisable.

French chemists made a special effort this year to secure what they were pleased to term an International Congress for Chemists, which held its meetings in Paris from the 29th of July to the 4th of August, inclusive. Reports of their proceedings have not yet reached us, but their announcement as published in advance would indicate that the term "international" was probably a misnomer, and that it was composed almost, if not entirely, of French chemists. This organization proposed a very wide field of discussion, the whole programme of which is too long for me to notice in this connection; the first section, however, proposed to discuss and, if possible, arrive at some agreement upon the nomenclature of organic chemistry; the second proposed to consider the analyses of organic matter, embracing analyses of soils, fertilizers, and feed-stuffs. The third section proposed to discuss the analyses of foods and drinks, and the fourth section the analyses of pharmaceutical products.

For two years a committee of the American Association for the Advancement of Science has been laboring to secure some agreement among analytical chemists in this country upon some uniform method of analyzing potable water and of stating the results. Some progress has been made, but not enough as yet to secure uniformity.

A study of the results of the analyses of uniform samples sent out by this association has developed the fact that very much more depends upon the individual skill of the analyst and attention to seemingly unimportant details than is generally supposed. As might be expected, the personnel of the association is constantly changing, and with these changes analysts comparatively unfamiliar with the application of the processes of analyses are frequently introduced into the work. There are many difficulties connected with the analysis of agricultural products which have not been overcome, and which the experience of the past shows may still require much study to overcome, but we are confident that we are on the right road to accomplish the work.

Variations altogether too wide appear in the results of the analyses of the same sample by men working upon it in different sections of the country. The most that can be said is that analysis is an approximation to the truth, the closeness of which depends upon the skill and experience of the analyst. Every one is supposed to be working by exactly the same methods and conducting the work with the greatest possible care. Why such variation in results? We may not be able to answer this question to our own satisfaction, but we are quite confident that with the care taken by this association to secure perfectly accurate and reliable methods of analyses no one else will be able to find out the cause of this variation sooner than ourselves.

These meetings have the highest possible educational value upon the analysts of the country engaged in this work, and it is hoped that the utmost freedom of discussion will prevail.

Since our last meeting a tremendous stride forward has been made by our Government in the establishment of agricultural experiment stations over the country, supported by the national appropriations and also in some cases by State appropriations. There are now in this country forty-six experiment stations engaged in the chemical study of agricultural products. Each of these stations has at least one analyst. Some of them have three or four engaged upon the work. In addition to this several of the States support State experiment stations, and have a fertilizer control distinct from the stations. Our agricultural colleges also in many cases have analytical chemists engaged in similar work. We have, therefore, a body of men engaged upon this work which in numbers will compare favorably with the assembled agricultural chemists of the whole of Europe. The Government now supports the work liberally, public sentiment is in favor of it, and it remains with the chemists of the country to determine whether they will meet the expectations of the public.

Our system of investigation is imperial in its conception. It is the assault of an army of trained scientific men upon the problems of nature, from which we may expect great results and great benefits. We are pioneers, blazing out a new route through an unexplored wilderness. We may lose our bearings; we may make mistakes; many may fall out of our ranks; but we are marching forward to conquer, and conquer we will. This movement of the Government represents one of the great waves of scientific influence that sometimes sweeps over the country carrying everything before it. It is an epoch in the history of the advancement of agriculture, just as we have epochs in the development of other branches of science. It means the establishment of the oldest of all arts, and the most neglected, upon a sure scientific basis, which must lead to the material development of the country—to prosperity, wealth, and happiness.

I am proud that we are living in this age of division of labor, unity, of purpose, and combination of forces, and when the principle of military precision is being carried into every sphere of activity in life.

Mr. Scovell, on behalf of the executive committee, moved that a committee of three be appointed by the president upon order of business. The motion was seconded and carried.

The president appointed Messrs. Wiley, Scovell, and Harrington.

Mr. Richardson tendered his resignation as secretary of the association, but, upon request of the president, consented to act until a secretary *pro tempore* should be chosen to serve during the present convention.

On motion of Mr. Wiley, a recess of twenty minutes was taken in order to afford the committee on order of business an opportunity to prepare its report. Upon re-assembling the following report was read by Mr. Wiley, chairman of the committee:

PROGRAMME.

HOURS OF MEETING.—Morning hours, 9.30 to 12.30; afternoons, at 2 p. m.

1. Report of Mr. G. C. Caldwell on Analysis of Cattle Foods.
2. Discussion.
3. Report of Mr. Richard H. Gaines on Analysis of Phosphoric Acid.
4. Discussion.
5. Report of Committee on Organization of National Chemical Society.
6. Paper, by Mr. Atwater, on Lines of Investigations for Chemists.
7. Report on Analysis of Potash, by Mr. E. H. Jenkins.
8. Discussion.
9. Report on Determination of Nitrogen, by Mr. M. A. Scovell.
10. Discussion.
11. Report on Analysis of Dairy Products, by Mr. H. W. Wiley.
12. Discussion.
13. Report on Analysis of Fermented Liquors, by Mr. W. B. Rising.
14. Discussion.
15. Report of Mr. W. C. Stubbs on Analysis of Sugars.
16. Discussion.
17. Business meeting; election of officers, etc.

The committee announces that a short-hand reporter has been secured through the courtesy of the Acting Secretary of Agriculture to transcribe the discussions.

The committee recommends that each one should speak slowly and distinctly, since chemical terms are difficult to take in short-hand.

Papers on subjects reported to the association should be read during the discussions of the subject treated therein. No one should be allowed to speak twice on the same subject until others desiring to speak thereon shall have had an opportunity to do so.

H. W. WILEY.
M. A. SCOVELL.
H. H. HARRINGTON.

It was moved that the programme recommended by the committee on Order of Business be adopted. The motion was seconded and carried.

Mr. Caldwell presented the following report on Analysis of Cattle Food.

REPORT ON THE ANALYSIS OF CATTLE FOODS.

MR. G. C. CALDWELL, *Reporter.*

This report is given in three parts, as follows:

PART 1.—The journal bibliography of the subject up to August, 1889, not including the nitrogenous substance.

PART 2.—Report on the analyses of samples sent out for 1888–'89.

PART 3.—Method of analysis adopted for 1889–'90.

Part 1.

SUMMARY OF THE LITERATURE IN THE PERIODICALS ON THE METHODS OF ANALYSIS OF CATTLE FOODS, UP TO AUGUST, 1889.

[Arranged under the head of each constituent, in chronological order.]

THE DETERMINATION OF THE ASH.

Arendt and Knop. *Grasuntersuchungen.* Die Versuchs-Stationen, II, 1860, 37: Fifteen to twenty-five grams incinerated in platinum or silver dish, moistened several times with water, each time dried and ignited again; acknowledged not to be accurate.

W. Hofmeister. *Analytisches Verfahren bei der Uutersuchung der Futterstoffe, etc.* Die Versuchs-Stationen, VI, 1864, 496: Ignition in muffle furnace, and pure (CO_2 and C free) ash given.

G. Kühn and others. *Analytische Belege.* Die Versuchs-Stationen, XI, 1869, 200: Knop's method; substance burned over small spirit-lamp in shallow platinum dish.

L. Lenz. *Analysen von Getreidesamen und einigen Futtermitteln.* Die Versuchs-Stationen, XII, 1870, 544: The substance charred in a muffle, the char transferred to, platinum dish, and with aid of artificial circulation of air incinerated over spirit-lamp.

A. Bechamp. *Nouvelle methode d'incinération des matières vegétales et animales.* Comptes Rendus, LXXIII, 1872, 337: The author makes use of bismuthic nitrate, intimately mixed with the substance to facilitate incineration in difficult cases.

H. Bornträger. *Einfache und sichere Methode zur Einascherung der verschiedenen Mehlsorten.* Fresenius, Zeitschrift, XVII, 1878, 440: The substance is mixed with ammonium nitrate, a white ash obtained in fifteen to twenty minutes.

G. Kühn and others. *Versuche über die Verdaulichkeit der Weizenkleie, etc.* Die Versuchs-Stationen, XXIX, 1883, 15: The "Weende" method followed.

S. M. Babcock. *Analytical methods.* Second Report New York State Experiment Station, 1883, 163: Ignites to low redness, with platinum disk suspended close over the dish containing the substance; against the under surface of the disk a gas flame directed, heating it red hot. Very low lamp-flame under dish. Gives only crude ash.

H. P. Armsby. *Methods of analysis.* Third Report Wisconsin Experiment Station, 1885, 138: Two grams burned at low temperature, carbonic acid and charcoal deducted.

H. W. Wiley and H. Edson. *Determination of ash in molasses, honey, etc.* Journal of Analytical Chemistry, III, 1889, 24: Results of several trials with zinc oxide indicate that ordinary careful incineration in a muffle "is to be preferred to the methods depending upon the admixture of any other substance to be used as a carrier of oxygen."

THE ETHER-EXTRACT.

C. M. Eisenstück. *Beiträge zur Kenntniss der Futtermittel.* Die Versuchs-Stationen, III, 1861, 237: Treatment either with ether vapor for twelve hours, or by digestion with cold ether for one to two days, and, in the latter case, determination of the extract in an aliquot part of the whole volume of ether and substance.

W. Henneberg. *Analytishches Verfahren bei der Untersuchung der Futterstoffen, etc.* Die Versuchs-Stationen, VI, 1864, 497: Extraction by several portions of boiling, anhydrous ether, in flask with return-flow condenser, taking off solution each time by means of a wash-bottle arrangement, the mouth of the inner tube being covered with finest muslin. All these portions filtered, the whole made up to 500 cubic centimeters and extract determined in aliquot part.

L. LENZ. *Analysen von Getreidesamen und einigen Futtermitteln.* Die Versuchs-Stationen, XII, 1870, 344: The substance allowed to stand twenty-four hours in contact with ether in closed flask, then boiled several times with ether.

M. W. SIEWERT. *Zur Fettbestimmung durch Aether.* Die Versuchs-Stationen, XXIII 1878: Recommends carbon disulphide in case of oil-cake, because it is easily freed from impurities, induces no decomposition, oxidation, or etherification of the fat; extraction in four hours.

P. WAGNER. *Fettbestimmung in Handelsfuttermitteln.* Die Versuchs-Stationen, XXIV, 1879, 289: Compares results with absolute and ordinary ether, and air-dried and anhydrous or nearly anhydrous substance. With air-dried substance, containing 9.17 per cent. of moisture, and ordinary ether (.75 specific gravity), gets 13 per cent. of fat, and proves that all thus extracted was soluble in absolute ether. With substance dried at 100°, or over sulphuric acid and containing 1.07 per cent. of moisture, and absolute ether, gets 10.54 and 10.73 per cent. of extract. With air-dried substance and absolute ether, 10.74 per cent.

To explain this, the author takes the ground that the absolute ether renders the substance anhydrous, or nearly so. Asserts that the fat contained in palm-nut meal is inclosed in cells, and if the walls of these cells are dried the fat is not so easily extracted, and thinks the same is true for all fodders. Promises full particulars of his work, of which this is an abstract given at meeting of directors of Experiment Stations, 1879.

W. O. ATWATER. *The quantitative determination of fats.* Proceedings of American Association for the Advancement of Science, 1881, 357: Number of experiments adduced showing that much more is extracted by fat-solvent agents in the case of vegetable substances originally dried in hydrogen than if dried in air, and considered that a serious error may thereby be introduced in the latter case.

G. KÜHN, and others. *Versuche über die Verdaulichkeit der Weizenkleie, etc.* Die Versuchs-Stationen, XXIX, 1883, 15: Dried substance boiled six to eight hours with 500 to 600 cubic centimeters anhydrous ether, made up to 1,000 cubic centimeters, let stand twenty-four hours at room temperature, and fat determined in 500 cubic centimeters.

CLIFFORD RICHARDSON. *Investigation of the composition of American wheat and corn.* Bulletin No. 1, Chemical Division, Department of Agriculture, 1883, 18: The extraction was made either with ether or petroleum ether, either solvent giving the same result.

S. M. BABCOCK. *Analytical Methods.* Second Report New York State Experiment Station, 1883, 163: Dried the substance previous to extraction, at 100°, till weight practically constant; shows that prolonged drying at 100° (for several days) renders some of the fat insoluble.

Dr. VON WILM. *Ueber die Fettbestimmung in den Palmkernmehlen.* Die Versuchs-Stationen, XXXII, 1885, 1: Substance must be finely divided (staubfeine).

H. SCHULTZE. *Bericht der Sektion für landw. Versuchswesen der Naturforscher Versammlung zu Wiesbaden.* Die Versuchs-Stationen, XXXIV, 1887, 460: Notices wide differences in results on different lots of "Schlempe." Urges necessity of (1) flour-fine (mehl-feine) division of substance; (2) drying at exactly 100°; (3) extraction with absolute anhydrous ether: (4) drying of extract at exactly 100°. All who took part in the discussion agreed that these rules should be strictly adhered to.

L. BUHRING. *Ueber Fettbestimmung in Futtermitteln.* Die Versuchs-Stationen, XXXV, 1887, 421: Requirements, two to three hours previous drying of substance at 100°, the use of completely anhydrous ether, and extraction for ten hours. Original substance to be pulverized so that it will pass through .5 millimeter mesh.

M. MAERCKER. *Verhandlungen des Verbandes land. Versuchs-Stationen; report of committee.* Die Versuchs-Stationen, XXXV, 1888, 454: (1) The sample must be dried at temperature not over 100°; at higher temperature resinous substances liable to be formed; three hours' drying at 95° sufficient; but in case of oil-cakes more experiments needed on drying in hydrogen or in illuminating gas. (2) Absolute ether, free from alcohol, must be used, and it must be kept water free; upper end of return-flow condenser must be closed with a calcium chloride tube. (3) In case of mashes dried at 100°, use of hydrous ether gives higher result, and excess over yield by anhydrous ether found to be lactic acid.

P. BÄSSLER. Same reference as above. Drying in hydrogen essential for linseed cake; dried at 100° in air, results too low, possibly as much as 5 per cent.

THE FIBER.

E. N. HORSFORD. *Ueber den Werth verschiedener vegetabilischer Nahrungsmittel, etc.* Annalen der Chemie und Pharmacie, LVIII, 1846, 170: Fiber determined by digestion with dilute hydrochloric acid, renewed from time to time, and then with sodium hydrate; this continued for two months.

POGGIALE. Chemisches Centralblatt, 1859, 841: Believes that the successive treatment with acid and alkali always causes loss of cellulose by conversion into glucose; separates out the sugar and starch by boiling with 300 cubic centimeters of water, to which 6 cubic centimeters of strong hydrochloric acid added, and determines sugar in filtrate.

C. M. EISENSTÜCK. *Beiträge zur Kenntniss der Futtermittel.* Die Versuchs-Stationen, III, 1861, 237: Usual method; digestion with 3 per cent. hydrochloric acid, 3 per cent. sodium hydrate, nearly absolute alcohol and with ether, warming slightly. Only in case of oil-seed cake, previous extraction with ether; filtering of hydrochloric acid extract very tedious: only by use of pressure of an 8 to 10 foot column of water was he able to get filtration through in one day, in some cases.

V. HOFMEISTER. *Fütterungs-Versuche mit Schafen; Untersuchungs-Methoden, etc.* Die Versuchs-Stationen, VI, 1864, 325: Some experiments on behavior of paper cellulose with various reagents, and a large number of determinations, and conclusions as to method to be followed. Makes much account of previous treatment of substance with alcohol and ether, because filtration thus rendered much easier, especially if much fat present. Two hours' digestion with sufficient amount of 3 per cent. sulphuric acid at 80°, filtration and washing, and same with potassium hydrate solution; then digestion for one hour with 30 cubic centimeters concentrated acetic acid at boiling heat.

W. HENNEBERG. *Analytisches Verfahren bei der Untersuchung der Futtermitteln, etc.* Die Versuchs-Stationen, VI, 1864, 497: The original Weende method; 3 grams boiled with 50 cubic centimeters of 5 per cent. sulphuric acid, with constant replacement of the evaporated water; siphon off the extract, boil thirty minutes with 200 cubic centimeters of water, with replacement of evaporated water, and siphon off, and repeat this treatment with water; carry through similar set of operations with 50 cubic centimeters of 5 per cent. solution of potassium hydrate and with water, and collect residue on weighed filter. All siphoned liquids allowed to stand an hour for settling, siphon off clear alkaline liquid, and put sediment on above filter, wash as long as alkaline, then put acid sediment on same filter, wash with water, alcohol, and ether; dry, weigh, and determine ash.

H. SCHULZ. *Zur Kenntniss der Chicorie.* Die Versuchs-Stationen, IX, 1867, 215: Fiber determined by Grouven's method; digestion six hours with 5 per cent. sulphuric acid, washed residue dried, and digested in same manner with 3 per cent. sodium hydrate solution.

G. KÜHN, and others. *Analytische Belege.* Die Versuchs-Stationen, XI, 1869, 198: Henneberg's method followed, with slight modifications.

Discussion: Verhandlungen der sechsten Versammlung deutscher Agrikulturchemiker, etc., 1869. Die Versuchs-Stationen, XIII, 1870, 41.

STOHMANN. Schulte's method (maceration with potassium chlorate and concentrated nitric acid) gives no pure product of constant composition.

FRÜHLING. The Weende method yields no pure product, and with same sample can get constant results only by most careful maintenance of constant strength of solution, and equal times of treatment. Tried stronger acid and alkali without any gain in accuracy. No uniform results obtained with Schulze's method.

G. KÜHN. The object of the work not necessarily to get pure cellulose, but to establish a method that with both fodders and excrements will give uniform results.

W. PILLITZ. *Beitrag zur Analyse der Getreidesorten und deren Mehle.* Fresenius, Zeitschrift, XI, 1872, 51: The substance is heated with dilute sulphuric acid eight hours to 140°–145° in sealed tube; the insoluble residue is weighed as fiber, and sugar, representing also the starch and dextrin, determined in the solution.

F. HEIDEPRIEM. *Fütterungsversuche mit Schafen.* Die Versuchs-Stationen, XVI, 1873, 28: Not a sharp determination; results may differ 3 per cent. on same sample.

J. KÖNIG. *Die Bestimmung der Cellulose und ihre Mangel.* Die Versuchs-Stationen, XVI, 1873, 415: Especially on his indirect method of estimating pure cellulose. Chlorine water and hypochlorite treatment unsuccessful. As to fineness of material, best results if made fine enough to pass through 0.5-millimeter mesh, but thinks this impracticable, as material must be dried and reground many times to get all through, and recommends use of 1-millimeter mesh sieve.

G. KERN. *Die Bestimmung der Cellulose.* Journal für Landwirthschaft, XXIV, 1876, 27: That the reagents used, acid and alkali, do not leave the cellulose untouched.

H. MÜLLER. *Eine neue Methode der Cellulose-Bestimmung.* Centralblatt für Agrikulturchemie, VI, 1877, 273: Based on fact that bromine water does not alter cellulose noticeably, but attacks the other constituents readily, and makes them soluble in ammonia.

F. HOLDEFLEISS. *Eine abgekürzte Methode der Rohfaserbestimmung.* Landwirthschaftliche Jahrbücher, VI, 1877, Supplement-Heft, 103: The substance is put in a pear-shaped flask, the lower part of which is drawn out into a tube; the throat of this flask-shaped funnel is stopped by a plug of asbestos, and the tube is passed through the cork of a suction filtering-bottle; the substance being put in the flask-funnel, with the acid or alkali, the mixture is both stirred up and heated by a current of steam, and when the action is completed suction is put on and the filtration is made; treatment with alkali, and washing with alcohol and ether in the same flask-funnel without removing the substance from it.

PETER COLLIER. *Methods for analysis of grain.* Report of the Commissioner of Agriculture, 1878, 152: Used 5 per cent. acid and 2 per cent. alkali solutions; in other respects the usual methods.

W. HENNEBERG. *Ueber Rohfaserbestimmungen.* Die Versuchs-Stationen, XXIII, 1879, 66: That fineness of the material affects the results, and suggests that tests be made of the range of this effect by working on material of different degrees of fineness.

C. KRAUCH. *Ueber die Bestimmung der Holzfaser und deren Mangel.* Die Versuchs-Stationen, XXIV, 1879, 295; XXV, 1880, 22; XXVII, 1882, 387: Takes the residue left after treatment of the vegetable substance with cold and hot water, malt extract, alcohol, and ether, which gives no reaction for starch, and calls this "Grundsubstanz," which he considers as consisting essentially only of cellulose and of lignine (Holzsubstanz) with higher proportion of carbon than in pure cellulose; shows that some of this is dissolved by the 1.25 per cent. acid and alkali, and reckoned with the nitrogen-free extract to a large extent in the case of some fodders; shows also that treatment with the Schulte reagent gives no constant result for pure cellulose.

H. WATTENBERG. *Eine vereinfachte Methode der Weender Rohfaserbestimmung.* Journal für Landwirthschaft, XXVIII, 1880, 273: Shows difficulties and sources of error in the original Weende method, involving six times siphoning off and the clarification of liquids by settling; that what thus settles from the acid liquid and washings escapes treatment with the alkali; and each time variable quantities of acid and alkali left in the dish; boiling in an evaporator, it is difficult to decide with any accuracy how much water must be added from time to time to keep volume of the liquid constant. Author uses evaporators with blue mark burned in at the 200 cubic centimeter level; draws liquid off by suction through paper filter on gauze tied over mouth of funnel; gets nearly all of the liquid off and rinses any substance on the paper back into the dish again; uses new paper for each drawing off; boils once only with water, for second washing merely pours boiling water on. Can complete a determination in one day.

CLIFFORD RICHARDSON. *Investigation on the composition of American wheat and corn.* Bulletin 1, Chemical Division, Department of Agriculture, 1883, 18: Fiber determined by the usual method except that the length of time of digestion was two hours, and 5 per cent. acid and alkali solutions were used; the work being done on a steam bath, the temperature of the liquid never went above 95°. Results agreed with those obtained in comparison tests with actual boiling of the liquids.

S. M. BABCOCK. *Analytical methods.* Second Report New York State Experiment Station, 1883, 163: Modified Wattenberg method; filters through asbestos in throat of funnel.

From *Praktische Arbeiten, Versuchs-Station Insterburg.* Landwirthschaftliche Jahrbücher XII, 1883. Supplement-Band 2, p, 14: Results by Weende method too low, by other methods could get just as pure a fiber and twice as large per cent.

A. WIELER. *Cellulosebestimmung.* Die Versuchs-Stationen, XXXII, 1885, 360: Tests Schulte's method; results not concordant.

H. P. ARMSBY. Third Report Wisconsin Experiment Station, 1885, 134: Follows substantially the Weende method as modified by Wattenberg.

WOLDEMAR und VON KNIEREM. *Ueber die Verwerthung der Cellulose im thierischen Organismus.* Zeitschrift für Biologie, XXI, 1885 ,67: Before proceeding on a long investigation of this question of the digestion of cellulose by different kinds of animals, Henneberg's Weende method was carefully tested and compared with Schulze's and with Müller's. This last was rejected as unreliable; the other two methods gave results agreeing well together, but Schulze's method was not found to be any better and took more time. Henneberg's gave very good results if special pains taken to reduce the substance to very fine powder.

W. HOFMEISTER. *Die Rohfaserbestimmung und das Holzgummi.* Die Versuchs-Stationen, XXXIII, 1886, 153: Previous removal of the fat makes action of reagents easier, as well as the filtration. Tries use of hydrochloric acid and potassium chlorate, the result, a larger percentage of cellulose.

E. F. LADD. *Fodder analyses, crude fiber.* Fifth Report New York State Experiment Station, 1886, 375: Several different methods compared of the treatment with acid and alkali of the usual strength, and boiling for the usual time, involving in some methods longer contact with the reagents than in others; in some methods nitrogenous matter and ash, one or both deducted, in others not; influence of previous extraction of the fat tested; also Holdefleiss' method tested with unfavorable results, and also influence of degree of heat during the treatment.

T. B. OSBORNE. *Notes on analytical methods.* Report Connecticut Experiment Station, 1886, 158: To get more rapid filtration, fits the paper filter so that a rift will be formed in it when the suction is put on.

STARCH, SUGAR.

DR. KROCKER. *Bestimmung des Starkmehlgehaltes in vegetabilischen Nahrungsmitteln.* Annalen der Chemie und Pharmacie, LVIII, 1846, 214: By the amount of carbon dioxide set free by fermentation by yeast, after previous conversion into sugar.

G. DRAGENDORFF. Chemisches Centralblatt, 1862, 523: The dried substance heated with a 5 per cent. solution of potassium hydrate in absolute alcohol, eighteen to thirty hours to 110° in sealed tube, filters, washes, dries, and weighs; loss equals albuminoids, fat, sugar, and part of salts; heats residue with 5 per cent. acid till no iodine reaction for starch; loss of weight equals starch.

H. SCHULZ. *Zur Kenntniss der Chicorie.* Die Versuchs-Stationen, IX, 1867: Determines starch and sugars in the acid extract obtained in the determination of the fiber.

V. HOFMEISTER. *Fütterungsversuche mit Schafen,* Die Versuchs-Stationen, X, 1868, 318: Boils with dilute sulphuric acid (1:8) and 100 cubic centimeters distilled water, keeping volume of water constant, lets stand 12 hours at common temperature, filters, makes filtrate alkaline and determines sugar by Fehling's process. As some of the fiber may be saccharified by this method, determines fiber in original substance by his method, and also in the residue from this determination of sugar and starch, the difference giving saccharified fiber.

C. BRIMMER. Die Versuchs-Stationen, XXVII, 1881, 389: A determination by extraction of bran with water, and conversion of starch to sugar by treatment with acid, etc., gave very different values for two samples, which by the ordinary method gave nearly equal nitrogen-free extracts, viz: 27.93 and 53.63 per cent. by the above method, and 65.87 and 65.28 per cent. by usual method.

E. F. LADD. *Methods of analysis.* Sixth Report New York State Experiment Station, 1887, 462: The sugars are determined in the aqueous extract. For the starch the substance is treated with 4 to 5 cubic centimeters of concentrated hydrochloric acid in 150 cubic centimeters of water at 100° for twelve hours, and then at common temperature over night; sugar is determined in the solution. A large number of determinations of sugars and starch in fodders given.

J. M. BARTLETT. *Report of chemist; determination of sugar and starch in coarse fodders.* Annual Report Maine State College Agricultural Experiment Station, 1888, 207: Sugar determined in water extract, before and after inversion. Residue from this extraction boiled thirty minutes with water, and thirty minutes more with 2 per cent. HCl. solution, filtered solution evaporated to 200 cubic centimeters, heated three hours with 15 cubic centimeters HCl. 1.15 specific gravity, and sugar determined in this solution. This method gave several per cent. less starch than Ladd's method, in which the acid treatment is more severe; and shown that by such treatment the cellulose is acted upon.

HYGROSCOPIC WATER.

W. HENNEBERG. *Analytisches Verfahren bei der Untersuchung der Füttermitteln, etc.* Die Versuchs-Stationen, VI, 1864, 497: The original Weende method; substance dried at 100° to 110°.

F. ROCHLEDER. *Zur Elementaranalyse organischer Substanzen.* Journal für praktische Chemie, C. 1867, 251: Describes arrangement for drying in current of carbon dioxide or hydrogen, consisting of a copper drying bath through which a tube passes from side to side; into this the substance is put, in a boat, and the gas is conducted through.

H. SCHULZ. *Zur Kenntniss der Chicorie. Ueber die angewandten Methoden.* Die Versuchs-Stationen, IX, 1867, 213: A water drying bath described for drying at temperatures above 100°, and provided with safety-valve.

L. SCHOONBRODT. *On the influence of drying on the active principles of plants.* American Journal of Pharmacy, third series, XVII, 1869, 318: (Abstract.) As result of tests with a number of plants, concluded that dried plants never entirely represent the fresh—a part of the change consisting in the oxidation of the fixed principles, besides the volatilization of a part of the volatile constituents.

G. KÜHN and others. *Analytische Belege.* Die Versuchs-Stationen, XI, 1869, 196: Substance dried at 80°, then allowed to stand exposed to air till saturated with hygroscopic moisture, then a portion dried at 100° in hydrogen.

H. WEISKE. *Modificirter Apparat zur Trockensubstanzbestimmung im Wasserstoffstrom.* Die Versuchs-Stationen, XVII, 1874, 31: The detemrination of dry substance by heating in current of hydrogen, is one most frequently recurring in agricultural chemical analysis; gives simple form of apparatus for it.

A. GAWALOVSKI. *Apparat zur schnellen Wasserbestimmung in hygroskopischen organischen Substanzen.* Fresenius, Zeitschrift, XIII, 1874, 267: Apparatus described for carrying air heated to 100° in a coil of pipe in saturated salt-water bath through the substance, in a flask of peculiar construction that is not itself heated.

F. TSCHAPLOWITZ. *Bestimmung der Trockensubstanz unter Anwendung von Alkohol.* Die Versuchs-Stationen, XXIV, 1879, 47: Especially applied to fruits; the substance is cut into thin slices, and boiled several times with absolute alcohol to which some ether has been added, and afterwards treated and washed with alcohol alone, and dried; aliquot parts of all the liquids, mixed together, with which the substance has been treated, taken for determination of solids extracted.

G. KÜHN. *Untersuchungsmethoden.* Die Versuchs-Stationen, XXIX, 1883, 8: In the case of a long series of analyses in connection with digestion experiments, dries the substance in hydrogen.

H. P. ARMSBY. Third Report Wisconsin Experiment Station, 1885, 138: Dries at 110° in current of illuminating gas.

K. ULSCH. *Apparat zur Bestimmung der Trockensubstanz in Gerste und Malz.* Chemisches Centralblatt, XVIII, 1887, 869: (Abstract.) The substance is contained in a tube, perforated at the bottom, which is fitted by means of a cork into a slightly larger copper tube with a lateral orifice near the top. This copper tube is itself fitted into an air bath; suction is applied at the mouth of the innermost glass tube, causing air to enter at the lateral orifice of the copper tube, and to pass thence to the perforated bottom of the glass tube and through the substance.

W. P. CUTTER. *Determination of hygroscopic moisture in air-dried fodders.* Bulletin 6, Cornell University Agricultural Experiment Station, 1889: Drying at temperature of boiling water at 100°, and at 110°, in air and in hydrogen compared. Loss of weight, not due to hygroscopic moisture, proved, if heated, to 110°, even in hydrogen.

MISCELLANEOUS.

W. PILLITZ. *Beitrag zur Analyse der Getreidesorten und deren Mehle.* Fresenius, Zeitschrift, XI, 1872, 51: Process given for determination of moisture, soluble and insoluble ash, fat, soluble and insoluble albuminoids, starch, dextrin, sugar, cellulose and extractive matters.

PETER COLLIER. *Methods for the analysis of grain.* Report of the Commissioner of Agriculture, 1878, 152: Oils, sugars, albumin insoluble in alcohol, albumin soluble in alcohol, gum, cellulose, starch and ash included in the determinations.

CLIFFORD RICHARDSON. *Investigation on the composition of American wheat and corn.* Bulletin 1 Chemical Division, Department of Agriculture, 1883, 18: Attempted the determination of soluble albuminoids and of dextrin, besides the usual proximate constituents, but relinquished them as unprofitable, considering the labor involved and want of accuracy.

H. P. ARMSBY. *Errors of analysis.* Third Report Wisconsin Experiment Station, 1885, 135: Results of some calculations of the probable error of some of the more important determinations.

PART 2.

REPORT ON THE ANALYSES IN 1888-'89.

Of the Experiment Station chemists who, on application, consented to take part this year in the analysis of cattle foods, nearly all, 17 in number, have sent in reports. As in the preceding year, three samples, hay, bran, and cotton-seed meal, were sent out. They were powdered so that the whole would pass through a sieve with 36 meshes to the linear inch or about 1.3 meshes to the millimeter; with the samples blank forms were sent for reporting the results, both in the original substance as analyzed, and calculated to dry substance.

The method of analysis to be followed was given in the report of the meeting of this Association in August, 1888. In order that I might be fully informed as to any variations in the management of the details of the method, where variations would be likely to occur, the following series of questions was sent out some time later, but not till so long after a few of the chemists had completed their analyses that they were not able to respond more than partially.

DETERMINATION OF THE ASH.

Is the carbon dioxide subtracted in calculating pure ash ?

DETERMINATION OF THE ETHER-EXTRACT.

How long was the substance dried previous to extraction ?
Dried till weight constant ?
Dried in boiling water drying bath, or in air bath at 100° C. ?
Dried in air, or in hydrogen ?
Time of extraction of the fat ?
Ether used, absolute, or specific gravity 0.72 ?
Extraction continued till nothing further extracted ?

DETERMINATION OF THE FIBER.

Time from first contact with acid till boiling began ?
Time in contact with boiling acid ?
Time required for filtration and for washing ?
Amount of washing water used ?
Time from first contact with alkali till boiling began ?
Time in contact with boiling alkali ?
Time required for filtration and for washing ?
Amount of washing water used ?
Strength of acid and alkali determined by areometer, or by titration ?
A brief description of material and arrangement of filter ?

The results of the analyses as calculated to the dry substance follow, together with the averages on each sample, the extreme limits of variation on each substance, and the extreme variation from the average in the case of each substance. I have, further, calculated these variations in percentage of the average per cent. of constituent found, in the case of the ether-extract, crude protein and crude fiber; this method of comparison, made use of now for the first time, so far as I am aware, brings out in a striking manner the relative extent of these variations, or errors in the work, inevitable or otherwise.

SAMPLE 1.—*Hay.*

Name of Station.	Moisture.	Composition of dry substance.						
		Pure ash.	Ether-extract.	Crude protein.	Crude fiber.	Nitrogen-free extract.	Total nitrogen.	Alb. nitrogen.
Alabama	10.79	*7.67	6.35	10.81	30.74	44.42	1.73	1.63
Connecticut	6.22	5.49	2.81	12.46	27.32	51.90	2.00	....
Illinois	11.62	*7.37	5.61	12.45	28.30	46.27	1.96	1.82
Maryland	8.55	5.48	3.53	13.87	27.91	49.21	2.22	2.03
Massachusetts	7.35	6.23	5.83	12.01	27.10	48.83	1.92	1.65
Michigan	6.60	6.23	4.42	13.12	27.69	48.54	2.10	1.86
Mississippi	10.48	*7.66	3.73	12.77	30.93	44.97	....	....
New Hampshire	8.78	5.66	4.39	12.85	27.75	49.35	2.07	1.79
New York:								
Cornell	8.57	5.66	5.40	12.50	28.90	47.54	2.00	1.93
Geneva	....	*7.06	4.60	12.31	28.84	47.19	1.97	1.84
North Carolina	10.59	6.31	7.10	12.68	27.67	46.24	2.04	1.70
South Carolina	10.10	*7.81	4.75	12.81	27.13	47.49	2.05	1.87
Texas	9.61	*7.91	6.89	12.72	27.44	45.04	1.84	....

*Crude ash.

SAMPLE 1.—*Hay*—Continued.

Name of Station.	Moisture.	Composition of dry substance.						
		Pure ash.	Ether-extract.	Crude protein.	Crude fiber.	Nitrogen-free extract.	Total nitrogen.	Alb. nitrogen.
Vermont	8.37	*7.88	5.33	13.27	30.11	43.40	2.12	1.78
Kentucky	8.68	6.94	3.03	12.65	27.87	49.51	2.03	1.84
Kansas	10.27	*7.76	2.66	12.57	28.23	48.78	2.01	1.70
Pennsylvania	7.03	*9.42	4.03	12.91	29.52	44.12	1.92	1.66
Average	8.98	*7.84 6.00	4.78	12.63	28.44	47.21	2.00	1.80
Difference between highest and lowest per cent	....	....	4.44	3.06	3.83	....	....	....
This difference expressed in per cent. of average per cent. of the constituent	....	....	96.79	24.23	13.47	....	....	....
Difference between average per cent. and the widest variation from it	....	....	2.32	—1.82	+2.49	....	....	....
This difference expressed in per cent. of average per cent. found	....	....	48.53	14.41	8.75	....	....	....

* Crude ash.

SAMPLE 2.—*Bran.*

Name of Station.	Moisture.	Composition of dry substance.						
		Pure ash.	Ether-extract.	Crude protein.	Crude fiber.	Nitrogen-free extract.	Total nitrogen.	Alb. nitrogen.
Alabama	10.78	*6.32	6.23	15.93	10.23	61.30	2.56	2.41
Connecticut	7.98	7.46	4.17	15.96	10.31	62.10	2.56	....
Illinois	11.23	*6.53	4.84	17.39	8.83	62.41	2.78	2.42
Maryland	9.90	7.72	4.26	16.90	11.83	59.29	2.71	2.59
Massachusetts	8.24	6.60	4.95	17.23	7.27	63.95	2.76	2.46
Michigan	7.60	6.67	4.37	17.56	8.22	63.18	2.81	2.30
Mississippi	10.45	*6.69	3.98	18.59	11.33	60.36	....	....
New Hampshire	9.56	7.33	4.55	15.98	10.65	61.49	2.55	2.39
New York:								
Cornell	8.83	6.69	4.88	16.97	9.72	61.54	2.71	2.39
Geneva	....	*6.24	4.76	17.25	9.07	62.68	2.76	2.36
North Carolina	11.55	6.75	5.33	17.49	8.48	61.95	2.80	2.35
South Carolina	10.59	*7.09	4.89	17.38	8.10	62.55	2.78	2.58
Texas	9.87	*6.41	5.04	16.64	9.00	62.91	2.40	....
Vermont	8.73	*6.47	4.96	17.63	8.93	60.01	2.82	2.59
Kentucky	9.08	6.53	4.51	17.25	8.52	63.19	2.76	2.46
Kansas	9.19	*7.39	4.03	15.83	11.44	61.31	2.53	2.03
Pennsylvania	7.45	*7.37	4.45	16.81	8.62	62.75	2.49	2.36
Average	9.42	*6.87 6.97	4.72	16.99	9.44	61.98	2.66	2.41
Difference between highest and lowest	....	....	2.25	2.76	4.56	....	....	....
This difference expressed in per cent. of average per cent	....	....	47.67	16.24	48.2	....	....	....
Widest difference from average	....	....	+1.51	+1.60	—2.39	....	....	....
This difference expressed in per cent. of average per cent	....	....	31.99	9.40	25.3	....	....	....

* Crude ash.

SAMPLE 3.—*Cotton-seed-cake meal.*

Name of Station.	Moisture.	Composition of dry substance.						
		Pure ash.	Ether-extract.	Crude protein.	Crude fiber.	Nitrogen-free extract.	Total nitrogen.	Alb. nitrogen.
Alabama	7.51	*7.27	12.92	50.74	3.89	25.19	8.12	7.88
Connecticut	6.75	7.17	11.16	53.96	3.54	24.17	8.63	....
Illinois	8.03	*7.59	11.39	54.25	3.71	23.06	8.68	8.42
Maryland	6.63	7.02	11.24	57.15	3.69	20.90	9.14	8.58
Massachusetts	5.96	6.99	11.95	53.64	3.21	24.21	8.58	8.25
Michigan	5.53	7.47	11.40	53.87	3.55	23.71	8.62	8.35
Mississippi	6.82	*7.62	11.09	57.98	4.16	25.19	....	....
New Hampshire	6.84	6.96	11.17	55.58	3.42	22.87	8.88	8.60
New York:								
Cornell	7.95	6.93	11.73	53.91	3.45	23.98	8.62	7.84
Geneva	....	*7.14	11.44	52.81	4.15	24.46	8.45	8.04
North Carolina	8.37	7.91	12.46	53.65	3.37	22.61	8.59	8.25
South Carolina	7.30	*7.81	11.90	53.48	3.17	23.63	8.56	8.30
Texas	6.09	*7.59	12.17	51.38	3.82	25.04	7.72	....
Vermont	6.39	*7.31	11.52	54.39	4.17	22.64	8.70	8.30
Kentucky	6.78	7.63	11.03	54.10	4.26	23.98	8.66	8.49
Kansas	7.10	*7.82	10.78	52.99	3.37	25.04	8.48	8.02
Pennsylvania	5.78	*7.80	11.19	63.66	3.94	23.41	8.09	7.83
Average	6.87	*7.59 7.25	11.56	53.97	3.72	23.83	8.53	8.22
Widest difference, highest and lowest	....	....	2.14	7.24	1.09	....	....	....
This difference expressed in per cent. of average per cent	....	....	18.51	13.41	29.03	....	....	....
Widest difference from average per cent	....	....	—1.36	+4.01	—.55	....	....	....
This difference expressed in per cent. of average per cent	....	....	11.76	7.43	14.78	....	....	....

* Crude ash.

The most significant comment on some of these series of results is presented in the percentage calculations at the bottom of each table. The results taken as a whole doubtless represent the average quality of the analytical work in the Experiment Station laboratories of this country. Some of them were obtained by chemists of much experience in this line of work, and others by chemists of little experience; for aught that we know the work may have been as well done as similar work is done in laboratories abroad. I am not aware that this work has been subjected to any such tests as these which we are applying to ours.

But I doubt if any member of the Association will be inclined, after even a brief consideration of these percentage statements of the limits of error, to regard our work as finished.

For comparison, in the following table the results are given of similar calculations applied to some analyses in other directions. Taking some determinations of total phosphoric acid reported at the convention of this Association in 1887, the results of which were declared by the chairman of the committee to be "in the main satisfactory," I get the following results on the limits of error calculated on per cent. of substance found. I give also a similar statement with respect to some of the determinations of potash reported at the same time, selecting the best series of results, a medium case, and the worst series; of this series the chairman, Mr. Richardson, says: "The averages may fairly be considered as correct, or close approximations to the true amount of potash in the specimens." But "the results, while showing close agreement among a few of the analysts, contain figures in other cases which are far from what they should be," and "the extremes which are so far apart occur in only two or three instances."

Designation of analyses.	Number of analyses reported.	Average per cent. of substance found.	Extreme limits of error calculated in per cent. of substance found.	Widest variation from the average calculated in per cent. of substance found.
(1) P_2O_5, total, 1887	6	12.84	9.27	6.53
(2) P_2O_5, total, 1887	6	12.27	9.14	6.77
(3) P_2O_5, total, 1887	6	19.51	2.71	1.38
(4) K_2O, 1887	4	53.83	0.67	0.39
(5) K_2O, 1887	12	13.58	5.82	3.1
(6) K_2O, 1887	8	13.49	8.45	4.67
(7) K_2O, 1887	9	2.93	19.45	11.61

It seems to me that we ought not to remain satisfied with our work as an association of official agricultural chemists till, for the analyses of cattle foods as executed by those who are responsible for such analyses at our Experiment Stations, the figure for the extreme variations in a series of analyses performed by different chemists on the same sample does not exceed for any constituent 15 per cent. of the average result of all the determinations of that constituent, or the widest variation from the average result does not exceed 10 per cent. of that average. It is evident enough that some vastly better work must be done than hitherto, to meet even this liberal requirement; it is evident, too, that it need not be by any means so liberal for some of the constituents; and it is certainly much to be desired that we may be able in time to work up to narrower limits than these. Unless by further effort closer agreement between results by different analysts can be assured than such as are exhibited in this report, we might perhaps as well give up making any further analyses of cattle foods, and fall back on the averages of the large number of analyses already made, as was suggested by the editor of Agricultural Science in a recent reference to this matter.

PART 3.

It remains now to be seen whether any practicable suggestions can be made as to the future course to be taken in a united effort for this closer agreement in results. Whatever suggestions I can offer will be based in part on a review of the replies sent in to the series of questions given on page 23. For this purpose I have prepared a careful summary of these replies. It does not appear to me necessary to include the replies themselves as a whole in my report. I shall also take into account the more recent journal literature of the subject so far as I have been able to trace it, and any suggestions that have come to me in private communications.

I shall take up each constituent by itself in the first place, and finally set forth the proposed course of analysis of cattle foods for the coming year, to be modified as the Association shall see fit.

The moisture.—No questions were put in regard to the determination of this constituent, except in connection with the preparation of the substance for the ether extraction. But reference to the best authorities and practice satisfies me that the desiccation should be performed at or not above 100°, in a current of hydrogen.

The ash.—The official method, giving pure ash, has, I regret to say, not been followed in all cases, more than half the analysts reporting only crude ash. This would be a matter of small account if it were not that one of the most important constituents, the nitrogen-free extract, has the misfortune to carry the load of all the errors made in the determination of the other constituents; by neglecting to deduct the carbon dioxide in the ash of the hay, and reporting only crude ash, the per cent. is 1.8 greater than it should be, and in the case of the bran 1.00 greater, according to special determinations made by my assistant; and the per cent. of nitrogen-free extract is correspondingly too low. I am not aware that carbon dioxide is allowed by any one as a normal constituent of the ash, The official method calls for the subtraction of the insoluble matter (sand and charcoal). It has been very properly noted by a member of the Association that a part of this insoluble matter may be silica that formed an integral part of the plant, which cannot, except with great difficulty if at all, be sharply separated from silica originally adhering as dirt. As for the subtraction of charcoal, the method of incineration recommended should leave none. Thus carbon dioxide is left as the only constituent of the crude ash that should unquestionably be subtracted.

In case the carbon dioxide is determined by absorption in potash solution, great care must be exercised that the gas is fully dried before it is carried into the potash bulb. Some experience in this regard in my laboratory has shown that such complete desiccation is not always as easy as one might suppose it to be, and that too high a percentage of carbon dioxide may be obtained, and consequently too low a result in pure ash.

The ether-extract.—If the necessity of uniformity in methods of analysis means anything, we need not be so much surprised by the percentage of error in this determination if we consider the want of this uniformity. The time of drying the sample previous to extraction ranged from thirty minutes to twenty-nine hours; the substance was in most cases dried to constant weight, but not in all; in some cases it was dried in a boiling-water bath, in others in an air-bath at 100°, in some cases in hydrogen, in others in air; the time of extraction ranged from four to nineteen hours; the ether used ranged from simple concentrated ether to absolute; the extraction was in nearly all cases continued as long as anything was extracted.

In the case of the hay, on which sample we have that enormous percentage of extreme limits of variation as calculated on one hundred parts of constituent found, the lowest ether-extract was obtained when all the directions of the official method were followed except as to time of extraction, only four hours; and the substance was originally dried in hydrogen, as well as, I believe, the extract; the highest per cent. was obtained with the use of simple concentrated sulphuric ether, but with very careful drying of the original substance; all the determinations of ether-extract at this Station were high, pointing to the kind of ether used as the probable cause of the results. Almost without exception the results on all the samples said to be obtained with absolute ether are below the average for each sample; and those who used Squibbs's stronger ether in almost all cases reported results above the averages. Other definite and frequently-occurring relations between results and methods of manipulation I am unable to discover, except that all results obtained by one chemist who dried his sample only thirty minutes previous to extraction and used Squibbs's stronger ether besides, are uncommonly high.

The points specially insisted upon in the more recent journal literature in regard to the determination of the ether-extract are, (1) uniform and fine pulverization of the substance; (2) careful drying in hydrogen and at a temperature not to exceed 100°; and (3) the use of anhydrous and alcohol-free ether. At the last meeting (1888) of directors of German Experiment Stations so much stress was laid on this matter of anhydrous ether, that it was agreed that the outer end of the return-flow condenser should be closed with a calcium chloride tube. If the exclusion of water is so important as is thus indicated, we should in our own work at least set out with an anhydrous reagent.

Less stress was laid on time of extraction, it being assumed, probably, that this would be continued as long as anything is extracted. I do not believe that reasonable uniformity in results will be attained till there is uniformity in manipulation as to the three points mentioned above.

In consideration of the difficulty in getting anhydrous and alcohol-free ether, it is well, in my opinion, to consider the advisability of substituting petroleum ether for sulphuric ether.

The crude fiber.—This determination, which all of us would be glad to eliminate from our work if it were possible, comes out much as might be expected in the percentage statement of limits of variation. The showing is much the worst in the bran, but it is not so very bad in the case of the hay. The manipulation by the several analysts was tolerably uniform: the chief differences were in the time required for filtration, and the consequent differences in time of exposure of the substance to the acid and alkali. The time required to bring the acid liquid up to the boiling point did not in any case except one exceed six minutes; in that one case ten to fourteen minutes were required. The boiling was in all cases continued thirty minutes. The shortest time required for filtration of the acid solution was half a minute, the longest six hours—on cotton-seed meal of course. The range of time required for the cotton-seed meal was remarkable. Two chemists completed the filtration of the acid extract in four and five minutes respectively, and the washing in fourteen and ten minutes; and the filtration of the alkaline extract was completed in one and a half minutes by one of these chemists and in twelve by another; the result obtained by one was somewhat above the average, and by the other a trifle below. The chemist whose acid extract required six hours for filtration, and the alkaline extract six hours more, obtained a result midway between the two above referred to. Another chemist, who reports a tedious filtration and washing, obtained a result a little above the average. It does not appear, from the comparison of results with varying time of exposure of the substance to the acid or alkaline liquid after boiling ceases, during the process of filtration, that this length of time has any uniform influence on the result; longer exposure does not, as might perhaps be expected, reduce the proportion of crude fiber found. This conclusion is in accordance with the result of special tests of this matter at the Connecticut Station, as well as with experience in my own laboratory.

A variety of filters was used. In general, but not always, those who attempted to use linen for the acid extract, as directed in the official method, had the most trouble as to the time required for the filtration and washing. At the Connecticut Station it is recommended in the use of paper filters to fit them loosely in such a manner that a rift will be formed down one side when the suction is put on; also, to shake the contents of the flask before throwing any of the extract on the filter, so as to mix up the coarse and fine material, and thus lessen the readiness with which the latter will get into and choke the pores of the paper. A felt of asbestos or of glass wool seems to have generally given much more easy filtration than the linen. Full instructions by which we could always secure such rapid filtration and washing, on oil meals, as was reported from the Maryland and Vermont Stations would be very acceptable to all fodder analysts.

The strength of the acid and alkaline solutions used was determined in some cases accurately by titration, in others simply by the areometer. I think that this should be done always by the more accurate method.

In the journals no part of the analysis of cattle foods has received more frequent notice than this, and it need not be said that it is regarded as an unsatisfactory determination; but I can glean only a little from these sources that will help us in our efforts to get more concordant results. Schulze's method, treatment with nitric acid and potassium chlorate, certainly gives no better results.

It is regarded as very important that the volume of the liquid should be kept constant during the boiling with the acid and alkali, which can be conveniently done by connecting the flask with a return flow condenser. This matter has not been properly noticed in our directions. Uniform and very fine pulverization of the substance is, of course, recognized as highly important for this as well as other determinations. König gets the most concordant results when the substance is all passed through a 0.5 millimeter mesh, but as this often requires such tedious grinding and sifting, he considers 1.0 millimeter as more practicable. One difficulty often encountered, the frothing during the boiling, which may sometimes lead to discordant results by removing a portion of the substance left adhering to the sides of the flask, from the thorough action of the reagent, is obviated at the Kansas Experiment Station by directing a blast of air on the surface of the liquid. Mr. Withers in my laboratory also originated the same device independently of Mr. Failyer, and used it with much success.

The crude protein.—Even in this determination, based as it is on an analytical process of acknowledged accuracy, the variations are in some cases discouragingly large, and show that the utmost care must be taken in the determination of the nitrogen, with scrupulous observance of all the directions given by the Association. Instead of sending to each chemist standard solutions made up in my laboratory, I sent out a small quantity of pure potassium tetroxalate prepared by myself, so that all the analysts could compare their solutions with reference to a common standard. This was far more convenient than sending the solutions themselves, and should in the hands of any reliable chemist answer as well.

Mr. Caldwell stated his opinion that in continuing the investigation of cattle foods the adopted method of analysis should accompany the samples sent with the request to take part in the work, which no one should engage to do who lacks facilities for scrupulously following the prescribed directions in every particular; that otherwise the value of the plan proposed can not be ascertained within the allotted time.

Mr. Richardson said that for years there had been no question in his mind but that all the analyses made in this country were more or less happy-go-lucky, the only comparable ones being those made by the same chemist. He believed that with the development of the experiment station system the time had arrived for a definite understanding as to what work should be done. Nothing, he urged, could more seriously damage the system than the accumulation of immense series of separate results which would never be compared; and this, he thought, was now a threatening evil. Many new stations have been and are to be established, with new laboratories and new workers, and it was within the power and the province of this Association to smooth the way by pointing out the mistakes which have been made by all its members and which are likely to be made by all beginners. He thought the suggestions of the reporter valuable, and recommended that they be adopted.

Mr. Caldwell said that, there being so large a number of chemists in this new field, many of them rather inexperienced, it seemed to him proper that the testing of their work by that of the Association should be made a part of the business of the stations, which ought to be considered as equally important with any of their other duties. He knew of nothing which would do more than that to encourage careful work.

Mr. Scovell, referring to Mr. Caldwell's report, recommended that in the determination of crude protein during the ensuing year the Kjeldahl method only be employed for cotton-seed meal, that method affording better results than are obtainable by the soda-lime method, which generally fails.

Mr. Caldwell said that the Kjeldahl method was the only one recommended in the report of last year.

Mr. Strahan said that in his laboratory the chief trouble had been met with in the determination of moisture; that it had been difficult to secure a full and sufficient supply of perfectly dry hydrogen in which to dry the samples for the proper length of time. A number of determinations had been made by that method, and it had occurred to Mr. White, the director of the laboratory, that ordinary gas, of which they had a good supply, could, if properly purified and dried, be used as a medium in which to determine the moisture. A simple arrangement had been devised to insure a constant supply of purified coal gas, and this was also used in heating the substance under examination. Ten or twelve determinations thus made as to hay, bran, and cotton-seed meal gave results exactly concordant with those obtained by the use of hydrogen. It was certainly more convenient to use the coal gas than to be under the necessity of preparing a current of hydrogen, and if no serious objections were raised it might be well to try and report upon the method described.

Mr. Atwater said that in many years of experience and hundreds of analyses and tests in his laboratory he had been unable to find that drying in hydrogen as compared with drying in air showed any such wide differences in the case of animal fats, mainly those in the muscular tissues of vertebrate animals, as were found in the case of vegetable substances; that nevertheless he had settled upon a uniform method of drying all substances in a current of hydrogen. An apparatus for this purpose which he described was nearly as convenient as the use of gas and perhaps not more expensive. In regard to vegetable fats, however, great differences were found. A sample of corn was dried in different ways and at different times. Dried in hydrogen to constant weight, the percentage of fat was something like 4.75, while dried in hydrogen not well purified, and especially when dried in air, the possible amount extractable with ether was reduced to somewhat less than 1 per cent. A similar experience was had with hay. The difference was attributed mainly to the oxidation of the fat.

At 12.30 a recess was taken until 2 o'clock.

AFTERNOON SESSION, TUESDAY.

The convention was called to order by the president at 2 p. m.

Mr. Jenkins moved that Mr. H. W. Wiley be made secretary *pro tempore*. The motion was seconded and carried.

Mr. Jenkins said that in the analysis of cattle foods he considered it necessary in order to secure accuracy of result that the moisture be determined in hydrogen, its determination in a stream of air being objectionable on account of the liability of fats to oxidation, rendering them insoluble in ether. He considered 100° as the best maximum, having found volatile oily matter of some kind passing off when certain feeding stuffs were dried rapidly at 110° in a stream of air or any other gas. Of course any temperature was open to objection; starch could not be perfectly dried at 100° nor satisfactorily at any higher temperature; German chemists had carried the heat up to 120° and 130°, till the starch evidently decomposed, and the higher the temperature the greater had been the amount of moisture driven off. As to the ash, he agreed with Mr. Caldwell that carbonic acid should be determined and the insoluble matter left in the ash unless the material was evidently very sandy, in which case judgment should be used in separating that sand. It was not possible with a solution of sodium carbonate of any strength to make a good separation of sand from the silica forming an integral part of the plant, and in raw fodders the silica occurs constantly and in considerable quantity. As regarded the ether extract, he had dried ether over chloride of calcium, assuming that if it contained but a small amount of alcohol the calcium chloride would remove it, as it is well known to form a non-volatile compound with alcohol. It would seem worth while for the Association to work in this direction, to see whether a perfectly alcohol-free ether could be had. There was no need of modifying the method for determining nitrogen, the present one giving absolutely accurate results if scrupulously followed. With crude fiber the Weende method seemed the best for the present. In the case of cotton-seed meal his determinations had been unsatisfactory on account of the gummy nature of the substance; some of the determinations were not reported, it having been impossible to get satisfactory filtrations. The previous extraction with ether helped the crude fiber determination considerably by removing the fat, and some one had suggested that an extraction with alcohol was also helpful. The use of coal gas instead of hydrogen for drying did not seem objectionable, provided the coal gas could be thoroughly purified, but hydrogen was about as cheap and was thoroughly clean.

The president asked whether Mr. Jenkins had ever tried carbonic-acid gas.

Mr. Jenkins said that he had not tried it; that it was very difficult to prepare it cheaply free from oxygen.

Mr. Burney said that some one (he thought Mr. Jenkins) had recently published a bulletin containing the results of certain investigations on the determination of moisture, and that one of the methods stated to have been employed was the use of a vacuum and sulphuric acid or an atmosphere of hydrogen and sulphuric acid, not elevating the temperature at all. He would like to ask Mr. Jenkins about that.

Mr. Jenkins said that last year the reporter on cattle foods had given the results obtained in drying the fodders by a number of methods, including, possibly, the use of a current of air. Mr. Jenkins had tried that method and had given it up as unsatisfactory; he had also tried the determination of water in a vacuum, putting the substance in a small, strong bottle, which could be weighed and placed in a bath heated to 100° or 110°, but had found that, even at 100°, when the air was exhausted something besides water was removed; it could be seen in the exit tubes as an oily substance; the use of vacuum bottles was therefore abandoned. The oily substance referred to was investigated only so far as to determine that it was soluble in ether. A current of air would carry off substances which would not be removed by merely standing in the air.

Mr. Burney said that he did not mean a current of air; he did not see that a current of air would be any better than drying in the air even at 100; what he meant was a current of hydrogen—was that necessary? Could not the substance be dried in hydrogen at an ordinary temperature by allowing it to stand for, say, three or four days, or would not a vacuum and concentrated sulphuric acid answer the same purpose? From theoretical considerations he judged this to be doubtful, but if any investigations had been made along that line he should be glad to hear from them, for he did not wish to employ the current of hydrogen if the necessity for its use could be obviated. Hydrogen generators were soon exhausted, requiring renewal, and labor ought to be saved wherever possible without detriment to the analysis. The multiplication of details in manipulation was extremely objectionable, and the simplification of methods was certainly desirable when, as was frequently the case, the details of analysis had to be carried out by men whose experience was limited to three or four years, counting in their purely academic training.

Mr. Burney, having the floor, desired to say a few words with regard to the determination of crude fiber. He had noticed in examining the analyses presented by Mr. Caldwell that the results from his laboratory for crude fiber were slightly low, and in one or two instances, he thought, the lowest, a fact of which he was almost bold enough to be glad. He would like to be informed as to the kind of sodium hydrate that was used in making the standard solution employed by the Association in determining crude fiber. Was it ordinary sodium hydrate or chemically pure; and if chemically pure, what kind of chemically pure; was it chemically pure by alcohol, baryta, or sodium? Was it made from sodium? This seemed to him a matter of fundamental importance; any sodium hydrate on the market contained carbonate, and sometimes in very large quantities. It was not a matter of indifference whether the strength of the solution was determined by specific gravity or by titration; in neither case was the carbonate taken into account. Carbonate of sodium, he presumed, would act very differently from sodium hydrate in the determination of crude fiber. He would explain that he used sodium hydrate made from sodium and carefully preserved, air being cautiously excluded by sealing the bottle after the withdrawal of a portion, thus pains being taken to keep out carbonic acid, a precautionary measure which he thought ought not to be overlooked, as probably it frequently was; where one determined the strength of the solution by the specific gravity method rather than by a process of titration, he would hardly take the extra precaution of using sodium hydrate absolutely pure, or as nearly so as it could be obtained on the market. If it were necessary to be so careful in the determination of ash, to estimate pure ash rather than crude ash, then it was much more necessary to be careful in the determination of the crude fiber, for the error there would naturally be much greater; the use of impure sodium hydrate would lead to far greater errors in estimating the crude fiber than could arise from the determination of crude ash rather than pure ash.

Mr. Jenkins said that the method of determining water in fodder was, of course, a conventional method, and it could not be said that either the merely hygroscopic water or the water both hygroscopic and combined were determined by any method that might be chosen. In drying the fodder at room temperature over sulphuric acid the result would depend upon the strength of the oil of vitriol. Some fodders when thoroughly dried, even at 100°, and put over even strong oil of vitriol, would dehydrate it and gain weight. He agreed that great care should be used as to the caustic soda; he used a 98 per cent. caustic sold by the Troy Laundry Machine Company, which had been proven to contain but a very small trace of carbonate.

Mr. Wiley said that he wished to call the attention of the reporter and the members of the association to the fact that certain very important kinds of cattle food were not included in the scheme; he referred to the cereals; not a word was said as to the method for their analysis; would it not be well for the reporter to include them? While suggesting this for the consideration of the reporter—perhaps not at the present meeting, but at the next meeting—he would like to say that he thought it would be a good idea to use the petroleum spirit or ether, if the object in this extraction was only fat, and he believed that any one seeing a column headed "ether extract" supposed fat to be meant. It was clear to all present that petroleum ether extract represented the fat much more nearly than sulphuric ether, and he would illustrate that by reference to some figures lately obtained in the analysis of sorghum seed. He would say first that the petroleum spirit purchased was fractionated; the aim was to use a petroleum which boiled at 40° to 45° centigrade; by redistilling it was entirely freed from any substance which would be left; the fractionation of the material was regarded as of the highest importance. He would suggest the use of an extractor similar to one which he exhibited and explained. In order to show that petroleum would extract more than ether he would read from a paper giving the results of seventy-two analyses of this cereal [sorghum]. He read as follows:

Means of seventy-two analyses of sorghum seed.

	Per cent.
Petroleum spirit extract	3.85
Sulphuric ether extract	.63

Continuing, Mr. Wiley said that he had used Squibbs's best ether taking it directly from the can and using it without further preparation. He thought it would be well to recommend and require the use of petroleum boiling at about 40° to 45°, which had been fractionated by the analyst himself so as to free it of all the non-volatile substances, and thus avoid the many difficulties which have been presented, and he should like to see the method extended to include the cereals. An attempt to analyze these by the method used for cattle foods would result in failure; the starch would not be dissolved within the time specified by acid of the strength employed.

The president said it was hoped that the processes of analysis adopted by the association would be generally adopted by the experiment stations of the country. It seemed to him, therefore, that great care should be taken at this meeting to adopt a process which all stations could try during the coming year. He would be glad to have the Department of Agriculture, as indicated by its Assistant Secretary at the morning session, supply the stations with a suitable blank upon which could be entered the results of the determinations made, so that the association might have for comparison the analyses of chemists in all parts of the country. That would enable the association to start aright. It was hoped that the work now beginning would extend through many years, and the foundation laid should be of such a character as would admit of the comparison of analyses. It would be unwise to permit Maryland, Pennsylvania, and New York to report analyses made by three several methods; some one system should be adopted upon the line of which all should work alike. At the very best only approximations could be made, but he believed that with the assistance the Department of Agriculture could give them in this direction something could be accomplished which would be found of great value later. He thought the point made by Mr. Wiley in reference to the use of petroleum ether or spirit was a most excellent one. A majority of chemists understood the "ether extract" to mean fat; in fact a great many of the reports said "fat," but it was not fat and did not mean fat; it meant a good deal more.

Considerable discussion had been had over the drying of substances, and the objection had been raised that a great many persons were not provided with the proper apparatus for supplying a stream of hydrogen for constant use. That ought to be the very first point made by a station chemist; he should say, "If you expect me to do the work give me the needful apparatus." He should represent to the director, or other presiding authority, that it would be useless to pay him a salary for doing work requiring hydrogen tanks, gasometers, etc., unless such apparatus were furnished. Every chemist should insist upon being supplied with proper facilities or should refuse to undertake the work. The speaker was not sure that carbonic acid gas could not be used in drying. The acid could be bought in tanks at very low rates;

a can would last a year or two, and the gas was a merchantable article which could be sent to any part of the United States in cans; he had it shipped to his station from New York, and found that cheaper than to make it himself; it was canned by machinery, and could be drawn from a tank made of boiler-iron in as big a stream and with as much pressure as might be desired.

Mr. Frear said that he had noticed with much interest, particularly in the publications of some of the experiment stations for the last two or three years, a work which he thought should be recognized by all as important. It was a fact, as had been intimated by Mr. Wiley, that methods of analysis which gave so-called close results in terms of groups of crude materials gave no clue to the amounts of the several real nutrients present; it was essential that the results of analyses should be such as could be used after they were obtained. He had for the last few years paid a good deal of particular attention to the nutritive qualities of certain green foods, and in common with other chemists had used the ether extract, but never felt satisfied in using it as a basis of calculation for the nutritive values of the foods, and the question had arisen in his mind whether he should not have to find something better than that before he could do work which he could regard as closely satisfactory. It seemed to him important, in the case of cereals, to get the starch and sugar as nearly as possible by separate methods—methods designed for the purpose of separating those constituents, and then to compare the crude fiber method that is used upon those foods and see how the two worked together. Great care should be taken not to leave some starch and call it fiber, and not to take out too much fiber and be unable to account for it. He thought that before very long it would be found exceedingly desirable to improve upon the analytical work now being done for certain purposes; while the crude separation of proximate materials into five or six very vaguely defined groups might answer for some purposes, he felt very certain that it would soon be considered necessary to follow much further the line suggested by the reporter on cattle foods, with a carefully designed method of proximate analysis which should give a more complete separation of materials. He should feel very much disappointed if within the next four or five years a point had not been reached where, instead of being content with the present grouping, he would in studies of digestibility and nutritive value, be satisfied with nothing less than a complete investigation of the foods used and of the excrement.

Mr. Scovell said that he had made some experiments with absolute ether and with Squibbs's ether upon the samples of hay, bran, and cotton-seed meal. The absolute ether was made by allowing ether to stand first with caustic potash, then with water, and finally with sodium. He thus got an absolute ether, and in the case of hay, for instance, the extract was 3.03 per cent., apparently a very low result. By extracting for eight hours, allowing it to stand over night, stopped up, and extracting again the next morning with the absolute ether, a larger result was obtained. This was kept up until it was entirely exhausted. He then put on the Squibbs's ether, supposed to be at least anhydrous, but not alcohol-free, and the result after extracting for eight hours was 1.17 per cent. more of ether extract. The chlorophyl evidently was more fully extracted by the use of the Squibbs's ether, the can of which was opened for the first time when he used it as described. He got on the cotton-seed meal .48 per cent. more and on the bran .23 per cent., or *vice versa*—he had forgotten which. He therefore thought it would be of much benefit, as the reporter had said, to use for the next year either absolute ether or petroleum ether. He did not know whether Mr. Wiley had results where he used petroleum ether and afterward Squibbs's ether, but he believed that if absolute ether were used first and then Squibbs's ether the results would be higher.

Mr. Wiley supposed that there was no chlorophyl of any consequence in the sorghum meal used.

Mr. Scovell believed Mr. Wiley got .63 per cent. with Squibbs's ether after extracting with petroleum.

Mr. Wiley said that such was the fact.

Mr. Harrington moved that the recommendations of the reporter on cattle foods in regard to the scheme of analysis for the next year be taken up in order, read, discussed, and submitted to the convention for action. The motion was seconded and carried.

Mr. Caldwell said that as to moisture the only change recommended was that the substance be dried at 100° in hydrogen.

Mr. Harrington asked whether that meant 100° in the water-bath or in the air-bath?

Mr. Caldwell said that he meant exactly 100° in the air-bath.

Mr. Wiley said that he was sure it would be of interest to the members of the association if Mr. Caldwell would tell them what kind of apparatus he used and how he kept it at 100°; this would enable all to use the same apparatus. He exhibited and explained the apparatus used by himself.

Mr. Caldwell said that personally he had no satisfactory method of drying in hydrogen; it was a matter as to which he wished experiments to be made, at least in the way of getting suitable apparatus; he had proposed the change under discussion only because the matter was so universally recommended as to make it important to be decided. He believed that the German experiment stations had held last year that drying at 95° for three or four hours was sufficient for the preparation of the substance for extraction by ether, but was not sufficient for the determination of the moisture. As for himself, he had nothing to offer in that regard; he would acknowledge that it was something upon which he wished more information. He should say 100°; it might be a little below or a little above, but not more than one or two degrees. He thought it unquestionable that to go above that, making such a change as would render the fat insoluble, would affect the amount of fat that could be extracted.

Mr. Jenkins said that the apparatus used at his station consisted of a rectangular copper box, fitted with a condenser above to prevent loss of water. There was a hole for an accurate thermometer and he had made the solution boil at exactly 100° by putting in a little salt. It would be a work of great difficulty to make certain that the contents of the tube introduced into that chamber were just 100°, for the reason that the hydrogen passing through would cool it very slightly, but he believed that he got very near to 100°. His tubes containing the substances through which hydrogen was passed went through this bath inside a copper tube and were not wet. In his experience a glass tube in contact with boiling water for three hours lost weight sufficiently to vitiate an otherwise good result. He thought the glass containing the fodder, and which is to be weighed, should not be in contact with the solution. One point of caution worth mentioning was this: if one has to weigh many glass tubes he must be careful to let them rest for a considerable time after rubbing them; one or two persons in New York had ruined delicate and costly balances by putting upon them things recently wiped with a silk handkerchief; the balances became charged, and it had been impossible to discharge the electricity from them.

Mr. Wiley said that he always followed the directions given in bulletin No. 13, part 4. After wiping the glass thoroughly dry it was allowed to remain at least 15 minutes, sometimes longer, before weighing it, in order that any electricity developed on the surface might be discharged; but he was unable to see how a balance could be utterly and forever ruined in the way stated. It must be a new kind of Leyden jar that could not be discharged.

Mr. Atwater said that in his laboratory it had been found most convenient and satisfactory to use as a counterpoise a glass similar to the one containing the substance to be weighed. After subjection to the current the latter glass was taken out, stopped, and allowed to stand in the same atmosphere as that of the counterpoise until cool, when the stopper was removed, and the air within and without brought to an equilibrium. The stopper was then replaced, and the substance was again weighed, always with the counterpoise, the latter being a little lighter than either of the glasses used to hold the substance to be dried.

Mr. Caldwell asked whether Mr. Wiley did not get a temperature of 100°.

Mr. Wiley said that he did not; that at Washington the boiling point of water was not quite 100°, but it was considered near enough to 100°. He thought it would be well for every station to adopt the boiling point of water as being upon the whole the best temperature, as it was certainly the most constant. There could be no objection to drying at 95° in places where the moisture would go off at that temperature as easily as at 100°.

Mr. Caldwell said that he believed it would certainly save much trouble to adopt the uniform practice of drying at the boiling point of water, and would make that recommendation.

Mr. Burney said that one or two precautionary measures which had been mentioned should perhaps be insisted upon. If a current of hydrogen were to be used, the shape of the containing flask became a matter of importance. In an Erlenmeyer flask the substance to be dried would be exposed in the form of a broad thin film, and with variation of velocity in the current of hydrogen there would be no considerable fluctuation in the temperature of the thin film lying at the bottom of the flask containing the substance to be dried. It would be apparent to every one that the temperature of the hydrogen must vary very considerably with the velocity of the current. He could conceive that two men working side by side might dry at temperatures differing considerably according to the shapes of the containing glasses and the velocities of the currents of hydrogen. As he understood. Mr. Caldwell seemed to think that the temperature of the heated substance at least should be approximately 100° centigrade. It was rather a nice matter to produce the conditions which should raise the substance in the flask to 100°. Even the thickness of the walls in the glass itself was a matter of importance, and it was very necessary to take into account the thickness of the layer of substance.

Mr. Caldwell said that some one principle should be agreed upon and worked under. He thought that a small Erlenmeyer flask would be found very suitable for the purpose; he used very light ones; there was a good deal of surface; the current of hydrogen did not have to be very rapid; it might be very slow; he did not think that would affect the temperature. If this were adopted, the reporter could secure some form of apparatus in which the work might be conveniently performed. He would suggest an arrangement similar to that shown by Mr. Wiley. He considered that the important point to be agreed upon was whether to use 100° as the uniform temperature or adopt the plan of heating the substance to the boiling point of water.

Mr. Lupton said that he supposed all were agreed that 100° was the proper temperature, and although there might be practical difficulties in exactly controlling that temperature, he thought it would be well to adopt the recommendation of the reporter in that regard and heat as nearly as possible to 100°. He had no doubt but that some apparatus could be devised by which that temperature could be maintained; perhaps one like Mr. Wiley's would answer the purpose.

Mr. Engelhardt said that he could see no objection to heating the hydrogen up to 100° in boiling water, and the substance itself to the same point.

Mr. Scovell said that to adopt the boiling point of water as the standard temperature would test the thermometers; according to some that he had had the boiling point was 100°, and according to others it was lower.

Mr. Burney said that he had intended to suggest the point of heating the hydrogen itself to 100°. That, he thought, was too important a suggestion to be overlooked. No two chemists would use currents of the same velocity; the amount of hydrogen driven through the apparatus by one would differ from that driven through by another, and the influence of this fact would not be inconsiderable. This, of course, was theoretical, but it was only through theory that good manipulatory methods could be reached. If the hydrogen could be heated to 100° by a convenient process, he thought that should certainly be done.

Mr. Jenkins said that he agreed with Mr. Burney, and believed the heating could be readily accomplished by means of a block-tin pipe through the water-bath.

Mr. Richards said that the association would have enough to go on if the reporter, in making up his scheme, would say that the vessels should be within certain diameters, and that the stream of hydrogen should be run at a certain rate at a certain temperature, or within certain points, at the discretion of the analysts.

Mr. Scovell moved that in the recommendation of the reporter the words "boiling point of water" be substituted for the words "one hundred degrees," the former temperature to be the point at which the moisture of cattle foods should be determined. The motion was seconded and carried.

Mr. Caldwell said that the question as to hydrogen had not been passed upon.

It was moved that the recommendation of the reporter as amended, that the substance be dried in hydrogen at the boiling point of water, be adopted. The motion was seconded and carried.

It was moved that the recommendation of the reporter that the words "sand and charcoal" be omitted from the statement of method as now in force be adopted. The motion was seconded and carried.

Mr. Caldwell said that the object of the next recommendation was simply to call attention to the necessity of carefully drying the carbonic acid in determining the ash.

It was moved that the recommendation of the reporter that the carbonic acid in the ash be determined in a potash solution, after being carefully dried, be adopted. The motion was seconded and carried.

Mr. Strahan said that in connection with the matter of ether extract, which was the subject of the reporter's next recommendation, he wished to ask Mr. Wiley whether he had made any experiments with the petroleum spirit to test its extracting power in the presence of the natural moisture of the sample, and whether, under that condition, the fat would be extracted as thoroughly as when the moisture was out.

Mr. Wiley said that for quantitative purposes he had made no extractions except on the dried sample. He had, however, used petroleum spirit for extracting from bodies which contained so much fat that it was impossible to prepare them in any other way. A very small amount of fat was afterwards secured, and from this he judged that petroleum spirit would, in the presence of moisture, extract the greater quantity of the fat.

Mr. Harrington cited the statement of Dragendorff, in his work on plant analysis, to the effect that in certain cases where it is exceedingly difficult to dry a plant it is not needful to take out all the moisture when petroleum spirit is used. Mr. Harrington believed that for ordinary quantitative work, however, the sample should be dried.

It was moved that the recommendation of the reporter, as amended, that before extraction the substance be dried for four hours in a current of hydrogen at the boiling point of water, be adopted. The motion was seconded and carried.

Mr. Caldwell said that the next recommendation of the reporter was that the extracting agent should be anhydrous and alcohol-free ether; for that, to save time, he would substitute petroleum ether, which would answer the same purpose. He doubted, however, whether it was best to do this before experiments had been made, and thought it might be preferable to recommend anhydrous and alcohol-free ether for the coming year, with the understanding that the reporter should suggest and secure experiments by the chemists of some stations with petroleum ether.

Mr. Jenkins said that he should very strongly object to the substitution of petroleum ether for anhydrous ether. There were several reasons for this objection; one of these Mr. Caldwell had already alluded to. It was not exactly known how the two solvents would compare as to their solvent power on fodders of very different nature and composition. It was believed that petroleum ether would not extract chlorophyl, as anhydrous ether would, and that was a matter of importance as making a very considerable difference in the case of raw fodders. All the fodder analyses which had been made in this country and abroad were made with the use of ether as a solvent. All the work on rations for cattle had been done upon the basis of analyses in which what was called "fat" was not pure fat, but perfectly well understood to mean ether-extract. To take now another solvent, which acted on a certain class of fodders in a way very different from that of ether, would put the new work out of joint with all that had been done by experimenters heretofore and that was recorded in the existing mass of literature of cattle feeding. The work of the association should be so done that its results could be compared with those of European chemists, and the standards of digestibility, rations, etc., should not be rendered inapplicable to the future work of this country. He would strongly favor additional experimentation with petroleum ether, but not its substitution for anhydrous sulphuric ether, so called.

Mr. Wiley said that it was difficult to understand the food value of chlorophyl; he did not know that it had a very high value as a food stuff, but he saw no special reason for estimating it with the fat; he supposed that Mr. Jenkins would not deny that petroleum spirit took the fat out of these substances, and if that was not what was wanted the association should say so; on the other hand, if his idea was correct, and it was desired to remove the fat, then the cheapest and best material for taking it out should be used. So far as the analyses of feeding stuffs were concerned, he thought that after hearing the report of Mr. Caldwell the association should be more inclined to doubt the accuracy of those analyses than to accept them as standards for future work. He did not believe that the tables truly represented the amount of fat in the several substances, and if that determination were necessary to be made it should be made. Other substances were also extractable by ether. He had lately been led to suspect the existence of an alkaloid in cotton-seed meal, and had learned upon investigation that choline was present, and perhaps other bodies existing in considerable proportion, and largely extracted by the ether. While all were agreed that petroleum ether would remove the fat, he saw no reason for supposing that it would take out much else in appreciable quantity, and the object being the removal of the fat, that agent should be employed which would best produce the result. He was very much in favor of adopting at the present time the words "petroleum spirit" in place of the words "sulphuric ether."

Mr. Scovell said that he believed it would be a good plan to use both solvents, the dried substance to be first extracted with petroleum ether distilled at 40° centigrade, and the extracted residue to be further extracted by the absolute ether.

Mr. Frear said that such a plan would load the association with a great deal of work, which, though partly in the right direction, would not be economical on the whole. Personally he felt that the proposed substitution of petroleum ether for absolute ether was a step in advance; that there was no longer any warrant for grouping chlorophyl with fat in making analyses for the purpose of studying the nutritive values of foods; he would gladly begin now to use petroleum ether instead of absolute ether; while perfectly willing to agree to any suggestion looking to the successive study of the two solvents, he was unwilling to see the work of the association hampered by the necessity of making both extractions.

The president said that it was difficult to work with petroleum ether at 40° except in cold weather.

Mr. Wiley said that he had used it during the whole of the past summer, making hundreds of extractions ot sorghum flours without trouble. The substance was weighted down with wool, as was his custom with fine substances.

Mr. Caldwell said that he understood the proposed amendment to refer only to the samples sent out by the reporter, and not to the general method. The objection had been made that it would put a great deal of labor upon some of the stations, and it was suggested that the process under discussion be employed only for the reporter's samples.

Mr. Scovell said that he had not so stated it, but would be satisfied with that if it met with the approval of the majority; if the year's results showed petroleum ether to be the better agent, its use could be continued; if not, the chemists would have the results obtained by the other method to fall back upon.

Mr. Scovell moved that the recommendation of the reporter be so amended as to prescribe that the extraction be made first by petroleum ether at 40° or 45° centigrade and then by absolute ether. The motion was seconded and lost.

Mr. Jenkins moved that the recommendation of the reporter be so amended as to prescribe that the samples sent out by the reporter be analyzed according to the scheme proposed, and then extracted, first, by petroleum ether boiling at from 40° to 45° centigrade, and afterward by absolute ether. The motion was seconded and carried.

It was moved that the recommendation of the reporter, as amended, that the extracted matter be dried at the boiling point of water in a current of hydrogen, be adopted. The motion was seconded and carried.

It was moved that the recommendation of the reporter that, in determining the crude fiber, during the time of digestion with the acid and alkali the amount of liquid be kept constant, be adopted. The motion was seconded and carried.

Mr. Caldwell said that he would recommend that the liquid be kept from frothing by throwing a stream of air upon the surface; he thought that might affect the result by keeping the substance from getting on the side of the glass; he had found it very effective.

Mr. Harrington said that it seemed to him it would be difficult to keep the volume constant, particularly if a condensing tube were used.

Mr. Caldwell said that it had not been found difficult; he simply carried the air from a tank by means of a tube through the cork into the flask; a little below the flask the tube opened, and the current of air was drawn below the surface and passed out of the condenser; there was no difficulty in keeping the current sufficiently rapid to prevent the frothing; no particular pressure was required; the rapidity was somewhat dependent upon the pressure.

It was moved that the recommendation of the reporter that, in order to prevent the liquid from frothing, a current of air be blown upon it during the process of digestion, be adopted. The motion was seconded and carried.

Mr. Caldwell said that, having heard the remarks in regard to reagents, he would recommend that the acid and alkali be determined accurately by titration; that not more than half the members of the association had pursued that course during the past year.

It was moved that the recommendation of the reporter that the strength of the acid and alkali used in the process of digestion be determined by titration be adopted. The motion was seconded and carried.

Mr. Caldwell said that he would recommend that the alkali used be pure sodium hydrate; he thought this was a very important matter.

Mr. Burney said that he had always used sodium hydrate made from metallic sodium; it was not expensive, being so diluted that a very little of it went a long way, and even if it were expensive that consideration should give way to the point of accuracy. It was known that sodium hydrate made by sodium was approximately pure, as it was not, according to his experience, by alcohol. He was prepared to state positively that some which had come into his laboratory, marked "chemically pure by alcohol," was anything but chemically pure, being as vile stuff as any he had ever seen under that name. He wished to say, by way of emphasis, that too much stress could not be laid upon the character of the chemicals used; it was no wonder that varying results were obtained so long as comparatively little attention was paid to the chemicals bought upon the market. Any one who would test the so-called pure chemicals of commerce for one year would convince himself that in them there was a grave source of error.

Mr. Scovell said that he would prefer that the recommendation should read, "sodium hydrate, free from carbonate." His own most impure sodium had been that from alcohol, and the next that from sodium; finally he had taken the 98 per cent. pure (costing 6 cents per pound) and made a saturated solution; the carbonate remained undissolved and was decanted off, leaving it comparatively free from sodium carbonate.

Mr. Frear said that in his laboratory such matters were of decided importance, as he had to make a great number of determinations, and while willing to go to any reasonable expense to gain accuracy, he did not feel that greatly increased expense should be incurred on account of the small quantities of carbonate present in the alkali, or that the association should put upon itself the burden of using soda made from sodium.

Mr. Wiley said that what Mr. Burney had said in regard to the carbonate in the commercial article was very true. It would be remembered that two years ago he had reported to the association a sample of hydrate which he had used, containing 38 per cent. of carbonate; this, of course, was extraordinary. It was possible to get a good hydrate on the market by demanding it; last year he had sent back sample after sample, and finally got a hydrate with under 1 per cent. of impurity and good for all purposes; he supposed it would be as good for this work as for saponifications. He thought it hardly necessary to go to the unusual expense of getting hydrate from the metal when one that was perfectly reliable could be obtained otherwise.

Mr. Caldwell said that he would accept Mr. Scovell's amendment, and would recommend that sodium hydrate, practically free from carbonic acid, be used.

It was moved that the recommendation of the reporter, as amended, that the alkali used be sodium hydrate, practically free from carbonate, be adopted. The motion was seconded and carried.

Mr. Caldwell said that he had no further recommendations to present.

It was moved that the scheme of analysis recommended by the reporter on cattle foods, as theretofore presented item by item, and as modified by the several amendments proposed and made, be adopted as a whole. The motion was seconded and carried.

Mr. Gaines presented the following report on phosphoric acid:

REPORT ON PHOSPHORIC ACID.

In accordance with the constitution of the association the reporter on phosphoric acid herewith presents his statement of the work done. Following the custom heretofore adopted the report covers: (1) Brief notices of analytical methods proposed during the year for determining P_2O_5; (2) Results of work done by members of the association; (3) Recommendations for the next year.

The yellow precipitate of ammonium phospo-molybdate, is of variable composition. A. Grete (Ber. 21, 2762–2765; Abs. J. Soc. Chem. Ind., 7, 771; Abs. J. Chem. Soc., 1888, 1341), on attempting to find out the conditions under which a compound of constant composition is formed, observed that in the presence of gelatine molybdic acid gives at first a white flocculent precipitate which only on further addition of molybdic acid turns yellow. The great insolubility of this compound of phospho-molybdic acid and gelatine as well as its property of settling quickly caused him to base on this reaction a volumetric test for phosphoric acid. A known quantity of the solution is neutralized, using methyl-orange as indicator, and any precipitate which may be produced, dissolved by a few drops of nitric acid. An equal volume of a 75 per cent. solution of ammonium nitrate, about 1 gram of sodium sulphate, and 1 cubic centimeter of a solution of gelatine are added, the whole heated at about 80° to 90°, and titrated with a solution of ammonium molybdate until no further precipitation occurs. The mixture is constantly shaken during titration, and from time to time a little of the gelatine solution is added as long as it produces a precipitate. When the titration is almost completed, the solution is heated and stirred until the precipitate becomes granular; the end of the reaction is then easily observed. If too much molybdate has been run in, a few cubic centimeters of phosphoric acid can be added and the solution again titrated.

The analysis can be carried out in twenty to thirty minutes, and the results are very satisfactory. The solutions used are as follows:

(1) *Molybdic acid.*—One kilogram ammonium molybdate is dissolved in ammoniacal water and the solution mixed with a gelatine solution, made by dissolving 100 grams gelatine in water containing a little nitric acid and boiling for some time. The mixed solution is poured into about 3 liters of nitric acid, specific gravity 1.2, and then diluted to 10 liters and allowed to stand several days. A flocculent precipitate separates; after removing this, the solution keeps perfectly well.

(2) Ammonium nitrate solution containing 75 per cent. of the salt and about 40 grams sulphate per liter.

(3) *Glue solution.*—One hundred grams gelatine are dissolved in boiling water, to which about 50 cubic centimeters nitric acid has been added and made up to a liter; a few cubic centimeters of this solution are sufficient to precipitate 0.1 gram P_2O_5. If the gelatine solution contains phosphoric acid, as often happens, this must first be removed.

A. Stetzser (Chem. Zeit., 12, 492; Abs. J. Chem. Soc., 1889, 186) has found that the drawbacks to the method of estimating phosphoric acid in manures, etc., by direct precipitation with magnesia mixture in presence of ammonium citrate, are the length of time required for the complete precipitation, the inconvenience of the prolonged stirring, and the danger of scratching the sides of the vessel so that the ammonium magnesium phosphate becomes firmly attached to the glass. To avoid these difficulties he adds a small quantity of ashless filter-paper, pulped to a thick magma in ammonia, previous to stirring for five minutes with a mechanical stirrer; the ammonium magnesium phosphate may then be at once collected. In addition to the great saving of time, 10 beakers may be stirred at once.

G. Linossier (Bull. Soc. Chim., 50, 353–354; Abs. J. Chem. Soc., 1889, 308) proposes the following method for the estimation of phosphoric acid: The solution of the phosphate acidified with nitric acids and free from hydrochloric and sulphuric acids, is heated nearly to boiling and treated with an excess of bismuth nitrate. The clear, supernatant liquid is decanted on to a filter and the precipitate washed several times with boiling water; the filter is washed with a saturated aqueous solution of hydrogen sulphide, the mass of the precipitate treated in the same manner, and well shaken with the saturated hydrogen sulphide solution; the clear liquid is filtered and the residue treated with hydrogen sulphide. Finally the bismuth sulphide is put on to the filter and washed with the hydrogen sulphide solution until the filtrate no longer shows a reaction with Poirrier's orange. The filtrate is boiled and the phosphoric acid determined with decinormal soda solution, using Poirrier's orange 3 as indicator. For very exact determinations, a second flask of the same size and containing the same amount of water and indicator should be used for comparison.

H. Tzschucke (Zeit. Angew. Chem., 1888, 383–385; Abs. J. Chem. Soc., 1889, 439) describes the following method for the direct estimation of P_2O_5 as tricalcic phosphate:

To the phosphate solution there is added so much hydrochloric acid that calcium chloride produces no precipitate, then calcium chloride, and, lastly, an excess of ammonia. The mixture is immediately diluted with twice its volume of cold water, filtered at once on a rapid filter, and the precipitate washed with cold water. The excess of calcium chloride must not be large, and warming is to be avoided. Substances which already contain sufficient calcium have simply to be dissolved in hydrochloric or nitric acid, and precipitated by ammonia. If sesquioxides are present the phosphoric acid combined with them must be determined separately. In the case of impure substances the results are admittedly only approximate, but the facility of the determination gives it a certain value.

M. A. V. Reis (Zeit. Angew. Chem., 1888, 354–362; Abs. J. Chem. Soc., 1889, 439–440), gives an exhaustive review of the methods proposed for estimating P_2O_5 in basic slag. He has made comparative experiments on the following methods of effecting solution: Klein's fusion method (Abs. J. Chem. Soc., 1886, 740); Kosman's Aqua regia treatment (Abs. J. Chem. Soc., 1881, 489); treatment with nitric acid (Jeusch); Brunneman's process with Aqua regia and sulphuric acid (Abs. J. Chem. Soc., 1887, 527); Klein's, with hydrochloric acid (Abs., 1886, 835); the same as used by Thilo and by Keanlepohl without remoral of silica (Abs. J. Chem. Soc., 1888, 321); Vogel's cold treatment with hydrochloric acid; Müller's with hydrofluoric acid; Mohr's with dilute sulphuric acid (Abs. J. Chem. Soc., 1887, 864); and that of Ende and Loges, with concentrated sulphuric acid (Abs. J. C. Soc., 1887, 52).

The results of all these methods agreed within 0.15 per cent., showing that the simplest of them, the treatment with hydrochloric acid without removal of silica, may be adopted when the phosphoric acid is thrown down by molybdate.

Of methods for determinatien the following were tested:

A—Weighing as magnesium pyrophosphate after precipitation with molybdate.

B—Precipitation by molybdat at 90° from a solution containing 20 per cent. of ammonium nitrate and weighing, after drying, at 100 to 110.

C—Determination as molybdenum phospho-molybdate by igniting the precipitate thrown down at 40° (Meincke).

D—Korschett's potassium molybdate method.

E—Thilo's ammonia titration method (Abs. J. Chem., Soc., 1887, 526), which for convenience and rapidity the author prefers to all others where extreme accuracy is not wanted.

The most suitable indicator is azolithium. The change of color is gradual but the reddish violet tint corresponding to the point of neutrality is easily recognized after practice.

F.—Schindler's titration with lead acetate. (Abs. J. Chem. Soc., 1888, 753–757).

G.—Determination of ammonia in the precipitate, according to Isbut and Stutzer (Abs. J. Chem. Soc., 1888, 194).

K.—Mallot's uranium titration method (Abs. J. Chem. Soc., 1887, 1063).

I.—Mohr's uranium method.

L.—Direct precipitation by magnesia mixture, after adding citric acid (Abs. J. C. Soc., 1888, 991).

Following Vogel's instruction exactly the results came out too high in this last method, the precipitate being contaminated with silica, lime, and iron. The filtration, moreover, is tedious. The following improved process is recommended: 10 grams of the powdered slag is boiled with 50 cubic centimeters of hydrochloric acid; 50 cubic centimeters of concentrated sulphuric acid is then added, and the mixture heated until white fumes appear. After dilution it is again boiled till all the iron is in solution, then cooled and made up to 504 cubic centimeters; 32 cubic centimeters of filtered liquid is then precipitated according to Vogel's directions. Each centigram of pyrophosphate is equal to 1 per cent. of phosphoric acid. The whole determination requires two and a half hours.

M.—Precipitation by molybdate in citric acid solution (Werenskjold). The reagent used is prepared by dissolving 300 grams of ammonium molybdate to 1 liter, adding half a liter of 50 per cent. citric acid solution, heating, adding half a liter of nitric acid (1.4), boiling till any traces of phosphoric acid present are precipitated, and then oxidizing the reduced molybdic acid by a few cubic centimeters of hydrogen dioxide. The re-agent and the phosphate solution should be boiling when mixed, and the mixture kept for five or ten minutes at 100°. The weight of the precipitate mutiplied by 0.03933 gives that of the phosphoric anhydride.

N.—Ignition of the precipitate obtained as in B. The results are more regular than those obtained by drying at 100°. With the exception of D, G, and I, which are condemned, these methods all give fairly concordant results; L is the best for accuracy, E for rapidity.

O.—Lange (Chem. Zeit., 12, 1587, 1588; Abs. J. Soc. Chem. Ind.; Abs. J. Chem. Soc.) has made the attempt to employ the solution prepared according Kjeldahl's method for the estimation of nitrogen and phosphoric acid as well in organic bodies. Ten grams of the substance are transferred to a flask (300 to 350 cubic centimeters capacity), 50 cubic centimeters of concentrated sulphuric acid, and 0.5-1 gram of copper sulphate added. At first the mixture must be heated very carefully, and it should only be raised gradually to the boiling point when carbonization is complete. After two hours' (in exceptional cases, two and a half to three hours) vigorous ebullition the reaction should be complete, and after the precipitate has subsided, the supernatant liquid should be of a pure light-green tint. When cold the whole is rinsed into a 500 cubic centimeter flask, which has been previously half filled with water. The mixture is then cooled and made up to half a liter.

During ebullition the flask should occasionally be shaken about in order to wash down the small carbonized particles which adhere to the sides. The dilute solution is filtered, and 50 cubic centimeters of the filtrate (corresponding to one gram of the original substance) taken for the phosphoric acid estimation. For this purpose 100 cubic centimeters of "Märker's citrate solution" and 25 cubic centimeters of magnesia mixture are added, and the whole stirred up till precipitation sets in. The precipitate can be filtered off after a quarter of an hour. "Märker's citrate solution" is prepared as follows: One thousand five hundred grams of citric acid are dissolved in 3 liters of water and 5 liters of 24 per cent. ammonia and 7 liters of water added.

F. Seyfert (Chem. Zeit. 12, 1390, Abs. J. Chem. Soc., 1889, 548) has found that in the estimation of phosphoric acid in the presence of ammonium citrate the complete precipitation of ammonium-magnesium phosphate may be greatly accelerated by stirring twenty to thirty times with a feather with the barbs cut short, at the same time rubbing vigorously against the sides of the beaker. As soon as the precipitate has settled to some extent it may be filtered without pressure, and the whole analysis finished in a fraction of the time usually required.

John Hughes (Chem. News, 60, 91) points out the probable cause of differences in analytical results, on soluble phosphoric acid, for the same sample. Concentrated superphosphate containing a high per cent. of phosphoric acid soluble in water, usually contains in addition some phosphoric acid combined with oxide of iron and alumina in the form of precipitated phosphate. This precipitated or reduced phosphate, correctly speaking, is not soluble in water, but is soluble in a concentrated solution of the superphosphate. Therefore in extracting the ordinary soluble phosphoric acid in such superphosphates, or indeed any superphosphate made from materials containing iron and alumina, it is most important to take care that sufficient water is added for the *first extraction*, otherwise the soluble phosphoric acid is returned 1 or 2 per cent. too high. In other words, the less water used for the *first* extraction the higher the percentage of phosphoric acid reported.

RESULTS OF WORK DONE DURING THE YEAR.

Last December samples were sent to twenty official and commercial chemists with the request that the determinations be made during the first week in January. The samples were marked as follows:

No. 1. Guano.
No. 2. Acid phosphate.
No. 3. Ammoniated superphosphate.

The samples were very carefully mixed to secure uniformity—400 cubic centimeters of solution of ammonia citrate accompanied each lot of samples.

Reports were received at intervals from January to July from eleven laboratories, embracing the work of fifteen chemists.

One of the chemists who took part in the work reported that the citrate sent out was alkaline, while another reported that it was slightly acid. All of the solution was prepared at the same time and in the same vessels, great care being taken to secure absolute neutrality. The results obtained are given in the following tables:

No. 1.—*Guano.*

Analyst.	Moisture.	Total P_2O_5.
	Per cent.	*Per cent.*
L. M. Bartlett, Maine State College	14.10	22.50
D. H. Benson, Bradley Fertilizer Company		22.32
W. P. Cutter, Cornell University Experiment Station	16.02	22.36
F. B. Dancey, North Carolina Experiment Station		22.23
J. S. Fuelling, U. S. Department of Agriculture		22.03
Richard H. Gaines, Richmond, Va	13.72	22.72
E. H. Jenkins, Connecticut Experiment Station		22.37
H. B. McDonnell, Pennsylvania State College	13.39	22.73
L. H. Merrill, Maine State College	14.10	22.53
H. J. Patterson, Maryland Experiment Station		22.28
Shepard Laboratory, Charleston, S. C	12.97	22.65
Stillwell and Gladding, New York City	12.05	22.90
W. S. Sweetser, Pennsylvania State College	14.97	22.70
E. A. Von Schweinitz, U. S. Department of Agriculture		21.81
E. J. Willis, Richmond, Va		22.45

No. 2.—*Acid phosphate.*

Analyst.	Moisture.	Phosphoric acid.				Total. P_2O_5.
		Soluble.	Reverted.	Total available.	Insoluble.	
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
L. M. Bartlett, Maine State College	16.50	11.15	2.13	13.28	1.87	15.15
D. H. Benson, Bradley Fertilizer Company	15.77	11.18	1.94	13.12	1.95	15.07
W. P. Cutter, Cornell University Experiment Station	17.82	10.80	2.84	13.64	1.81	15.48
F. B. Dancey, North Carolina Experiment Station	....	11.12	2.00	13.12	1.56	14.68
J. S. Fuelling, U. S. Department of Agriculture	....	10.73	1.92	12.64	1.93	14.57
Richard H. Gaines, Richmond, Va	16.12	11.21	1.91	13.12	2.08	15.20
E. H. Jenkin's, Connecticut Experiment Station	....	11.21	2.05	13.26	1.46	14.72
H. B. McDonnell, Pennsylvania State College	15.62	....	....	13.49	1.62	15.11
L. H. Merrill, Maine State College	16.22	10.98	2.26	13.24	1.82	15.06
H. J. Patterson, Maryland Experiment Station	16.56	10.95	1.81	12.76	2.00	14.76
Shepard Laboratory, Charleston, S. C	16.16	11.01	2.25	13.26	1.71	14.97
Stillwell & Gladding, New York City	15.20	11.05	2.23	13.28	1.87	15.15
W. S. Sweetser, Pennsylvania State College	17.61	[1]10.86 [2]10.77	2.66	13.52	1.78	[3]15.28 [4]15.24
E. A. Von Schweinitz, U. S. Department of Agriculture	....	10.72	2.13	12.85	1.53	14.38
E. J. Willis, Richmond, Va	....	10.66	2.01	12.67	2.16	14.83

No. 3.—*Ammoniated superphosphate.*

L. M. Bartlett, Maine State College	10.38	6.98	2.02	9.00	1.65	10.65
D. H. Benson, Bradley Fertilizer Company	....	7.05	2.15	9.20	1.84	11.04
F. B. Dancey, North Carolina Experiment Station	....	6.96	2.13	9.09	1.69	10.78
J. S. Fuelling, U. S. Department of Agriculture	....	6.74	1.79	8.54	2.00	10.53
Richard H. Gaines, Richmond, Va	10.18	6.96	2.00	8.96	1.76	10.72
E. H. Jenkins, Connecticut Experiment Station	....	6.74	2.19	9.03	1.64	10.67
H. B. McDonnell, Pennsylvania State College	10.30	6.70	2.21	8.91	1.72	10.63
L. H. Merrill, Maine State College	10.49	6.82	2.15	9.07	1.66	10.73
H. J. Patterson, Maryland Experiment Station	12.16	6.76	2.12	8.88	1.72	10.60
Shepard Laboratory, Charleston, S. C	10.61	7.01	2.17	9.18	1.57	10.75
Stillwell & Gladding, New York City	10.20	6.97	1.70	8.67	2.02	10.70
W. S. Sweetser, Pennsylvania State College	13.65	[1]6.81 [2]6.83	2.08	8.89	1.92	[3]10.86 [4]10.87
E. A. Von Schweinitz, U. S. Department of Agriculture	....	6.63	1.90	8.53	1.62	10.15
E. J. Willis, Richmond, Va	....	6.61	2.02	8.63	1.93	10.56

[1] Washed by decantation.
[2] Washed on filter.
[3] Washed phospho molybdate with cold water.
[4] Washed phospho molybdate with ammonium nitrate.

Averages.

	Number of determinations.	Average.	Highest.	Lowest.	Difference.
Sample No. 1:					
Moisture	8	13.91	16.02	12.05	3 97
Total P_2O_5	15	22.43	22.90	21.81	1.09
Sample No. 2:					
Moisture	10	16.35	17.82	15.20	2.62
Soluble phosphoric acid	14	.10.97	11.21	10.66	.55
Reverted phosphoric acid	14	2.15	2.84	1.81	1.03
Available phosphoric acid	15	13.15	13.64	12.64	1.00
Insoluble phosphoric acid	15	1.81	2.16	1.53	.63
Total phosphoric acid	15	14.96	15.48	14.38	1.10
Sample No. 3:					
Moisture	8	10.99	13 65	10.18	3.47
Soluble phosphoric acid	14	6.83	7.05	6.61	.44
Reverted phosphoric acid	14	2.04	2.21	1.70	.51
Available phosphoric acid	14	8.89	9.20	8.53	.67
Insoluble phosphoric acid	14	1.76	2.02	1.57	.45
Total phosphoric acid	14	10.67	11.04	10.15	.89

Percentage of results differing from the average not more than—	.20.	.50.	1.00	1.50	2.00 & over
Sample No. 1:					
Moisture	37	37	62	75	100
Total P_2O_5	53	93	100	....	....
Sample No. 2:					
Moisture	30	50	70	100	....
Soluble phosphoric acid	57	100	....	....	....
Reverted phosphoric acid	53	87	100	....	....
Available phosphoric acid	53	93	100	....	....
Insoluble phosphoric acid	66	100	....	....	....
Total phosphoric acid	53	87	100	....	....
Sample No. 3:					
Moisture	....	12	75	87	100
Soluble phosphoric acid	85	100	....	....	....
Reverted phosphoric acid	85	100	....	....	....
Available phosphoric acid	57	100	....	....	....
Insoluble phosphoric acid	85	100	....	....	....
Total phosphoric acid	85	92	100	....	....

As will be seen from an examination of the tables the discrepancies in the determination of moisture are still very great although there is a marked improvement in this respect over the results of last year.

There is also more uniformity in the results on phosphoric acid. The greatest variation from the average in any case was 0.55 per cent.

The committee recommends a modification in the method for determining citrate insoluble phosphoric acid. After digesting with neutral citrate, filtering, and thoroughly washing, instead of incinerating filter and contents, drop the filter and contents immediately into 200 cubic centimeters flask, add 30 cubic centimeters of nitric acid and a little hydrochloric acid and digest over gentle heat until all organic matter is destroyed, then dilute to 200 cubic centimeters and proceed in the usual manner.

The committee recommends that the present general method of determining phosphoric acid be continued subject to such further modifications as the Association may deem advisable.

Respectfully submitted.

RICHARD H. GAINES.

Mr. Gaines said that he wished to add a few words of explanation and apology. He had conceived it to be his duty, as reporter on this important subject, to try the various methods proposed during the year and published in the foreign scientific journals; in American literature he had found only a few extracts from European publications. But one chemist was employed in the Virginia department of agriculture, and a vast deal of work devolved upon him; not until last Saturday had he succeeded in finishing the report presented. He would have liked to incorporate in the report tests of all the methods proposed in order that whatever in them seemed wise might be made use of in the work of the coming year, but the limited time at his disposal had not permitted this.

It was moved that the report be accepted.

A member asked whether that would involve the acceptance of the recommendations made by the reporter.

The president stated that it would not; the motion was merely preliminary, placing the recommendations in order for discussion.

The motion was seconded and carried.

Mr. Caldwell moved that the recommendation of the reporter be adopted. The motion was seconded.

At the request of the president, Mr. Gaines read the recommendation, as follows:

> The committee recommends a modification in the method for determining citrate insoluble phosphoric acid; after digesting with neutral citrate, filtering, and thoroughly washing, instead of incinerating filter and contents drop the filter and contents immediately into 200 cubic centimeter flask, add 30 cubic centimeters of nitric acid and a little hydrochloric acid and digest over gentle heat until all organic matter is destroyed; then dilute to 200 cubic centimeters and proceed in the usual manner.

Mr. Gaines said that the modification recommended was proposed to him by Mr. Dancy, of the North Carolina Experiment Station, whose letter containing the suggestion he had received last winter. He had at once tried the plan and found that it gave perfectly accurate results. At the time he had not noted down the figures, but he thought that the old plan of incinerating after washing afforded nothing preferable to the method now recommended, while it certainly involved a waste of time. The proposed plan dissolved out all the phosphoric acid. Mr. Dancy, who had tried it for some time, informed him that he found the results uniformly accurate. Mr. Battle had tried it, and would probably give the association the benefit of his experience.

Mr. Battle said that he could only verify the experience of Mr. Dancy; that incineration was entirely unnecessary, and was accompanied by liability of loss unless great care was used, whereas if the paper was put directly into the flask there was no such danger, and the nitric acid was as effectual as the heat in disintegrating the paper. He thought the recommendation was a very good one.

Mr. Kedzie asked whether the addition of a little chlorate of potassium would not be better than the nitric acid used to secure complete disintegration.

Mr. Gaines said that in his experience nitric acid had always proved effectual to make the reduction; the hydrochloric acid was added simply as a solvent to effect the solution of the matter that would not be attacked by the nitric acid.

Mr. Battle said that he had tried the chlorate of potash in connection with the nitric acid, and had found the nitric acid to be just as effectual without it, and had therefore omitted the chlorate of potash in his work.

Mr. Ross said that at the time when the magnesium and nitrate method was dispensed with he had made some experiments with regard to the use of chlorate of potash with hydrochloric acid for insolvent residues only. He supposed it to have been the original intention that the method of destruction of organic matter in the insoluble residue should be parallel with that used on the original fertilizer itself, and on all samples whose total phosphoric acid could be readily determined by the use of chlorate of potash along with hydrochloric acid he had found that equally good results could be obtained with the citrate insoluble residues.

Mr. Jenkins said that in such cases the actual analytical results should be placed before the association, upon which its action could be based. To those having official control of fertilizers any change in method was a somewhat serious matter, for every such change would be taken up by those people with whom the association was unpopular, and serve as a pretext for charging that the chemists were continually changing methods arbitrarily, and did not know their own minds. Personally, he was opposed to introducing an organic substance, and then depending on an oxydizing acid to remove it in the wet way. Although that might be all right in the present case, it was known that in other cases it was not all right; it was absolutely impossible to thoroughly destroy organic matter by treatment with nitric acid, the material would change its form and become colorless, but it was still there, as had been proven over and over again in toxical cases. In using the method proposed he should fear the presence of some organic acid that would play tricks with him afterward, but if burned with fire that was certainly the end of it. One could not throw a wet filter into a porcelain crucible, put a lamp under it, let it go, and get good results, but when a filter was pinched together at the top and was then laid bottom side up in the crucible and properly heated he did not see that there was any chance for the escape of phosphatic material; it seemed absolutely safe. In his experience there had not been the breakage of porcelain crucibles complained of by many. He would much prefer to keep on in the present way. When the question as to the solution of the phosphate should come up he would wish to say more upon the point of getting rid of the organic matter by oxidation rather than ignition. He preferred the present method for two reasons: First it was now in use, and was unobjectionable so far as accuracy was concerned, while the other method was a new one, and the association had not before it the results of experiments to prove its accuracy; and second, the absolute removal of organic matter in the wet way was not possible by the proposed method but was certain by the present method.

Mr. Gascoyne said that the adoption of the method recommended would be going back to the old method of four years ago, when the association had used the method of digesting the filter in nitric acid.

That method had been discarded. He had never found any loss by burning when carefully done; if the top of the filter was pinched before it was put in, there was no chance of losing anything.

Mr. Gaines said in sending out the samples he had added to the circular letter accompanying them a request that any other methods known to the chemists who participated in the work should be tried and their results reported. With only one exception the chemists had tried the method laid down in the bulletin, and had experimented on no other. Nearly all the members of the association were busy throughout the year, and perhaps it was too much to expect that any one would try a new method not universally adopted. To him the use of the method recommended had seemed so rational that he had tried it immediately, making half a dozen or more determinations; his assistants had also tried it, making duplicate work sometimes, and their agreements had been so close in all cases that he had abandoned the other method, had employed the one recommended in his own laboratory, and should continue to do so.

Mr. Caldwell moved that the recommendation of the reporter be adopted.

Mr. Battle moved to amend the recommendation so that the present method should stand as the official method of analysis, the method proposed by the reporter to be referred to in the letter accompanying the samples sent out next year, with the direction that it be tried, but no official change to be made until the reports should come in and be considered by the association at its next meeting.

Mr. Caldwell accepted the amendment.

The motion was seconded and carried.

Mr. Battle asked whether it would now be in order to refer to certain changes in the present methods of determination of phosphoric acid and moisture which seemed to him desirable.

The president stated that such questions were in order.

Mr. Battle said that, in regard to the preparation of the sample, it was not stated through what sieve it should pass, that matter being left open to choice; a 20-mesh, a 60-mesh, or a 100-mesh sieve might be used. In his own work he had found that a 20-mesh sieve would give a certain amount of citrate insoluble phosphoric acid, a 40-mesh sieve less, a 60-mesh sieve still less, and a 100-mesh sieve the least of all, so that it seemed absolutely necessary that a standard size of sieve should be specified for use in the preparation of the sample. He used invariably a 20-mesh sieve, but he wished to have some discussion of the matter, deeming it very important.

Mr. Frear said that he had some very trying experience during the past three or four months in getting results which would agree with those of other chemists, and in getting any two chemists to whom we had referred the matter to agree together as to certain samples of dissolved bone. He had adopted the general plan of letting the manufacturer mix and grind his goods, and his analyst, who determined the total phosphoric acid, had given him results which agreed quite well with the samples he had himself analyzed of goods turned out upon the retail market; therefore, for a short time, he had not prepared his own samples at all before analysis. But he had begun to hear from various quarters that his results were not altogether concordant with those of other chemists. He had at once written to these gentlemen to ascertain what methods they used, and had found that, while there was some difference, the majority of them used sieves of from 20 to 25 mesh. To determine so far as possible wherein the differences lay and what they were, he had sent out to a number of chemists two samples of what purported to be dissolved bone, with results which he would like to read, and which he thought would come in as well now as at any other time.

Two samples of dissolved bone were selected for test and marked A and B, respectively. For test methods, the following checks were made:

1. Determination of differences in effect produced by moderate and by hard rubbing in a beaker with pestle tipped with rubber, and in a mortar with bare pestle, using the sampler both before and after grinding and sifting through a 20-mesh sieve.
2. Test in the Pennsylvania Station, using Pennsylvania citrate and that prepared by Dr. Gascoyne.
3. Test in Connecticut Station, using Pennsylvania and Connecticut citrates.
4. Analyses following the association method as closely as possible, by six different chemists of experience.

All samples, except one, sent to chemists in other stations, were selected with extreme care, from the ground and sifted samples and inclosed in sealed tubes for transmission. The samples sent to Dr. Gascoyne were taken from the laboratory lot before grinding and sifting, but Dr. Gascoyne gave them a preparation similar to that mentioned above. The samples were necessarily very small, owing to the limited supply, but were as uniform as it was possible to make them.

The results are as follows:

	A.	B.
	Per cent.	*Per cent.*
Total P_2O_5.—(Determined by Mr. Frear, Pennsylvania Station)	18.20	19.25
In unground samples.		
Soluble and insoluble P_2O_5:		
By Dr. McDonnell, Pennsylvania Station, using Pennsylvania citrate, and rubbing *moderately* in beaker with rubber-tipped pestle:		
Soluble P_2O_5	5.71	7.97
Insoluble P_2O_5	7.31	7.85
Available P_2O_5	10.85	11.40
By Frear, same conditions as above, except that rubbing was *hard* for one and a half to two minutes each time:		
Insoluble P_2O_5	6.93	7.40
The results given in both cases are means of closely concordant duplicates.		
In samples after grinding.		
Soluble and insoluble P_2O_5 by Dr. McDonnell:		
Soluble:		
(*a*) Pennsylvania citrate, rubbing moderately in beaker with rubber tipped pestle	5.92	8.22
(*b*) Gascoyne's citrate, otherwise as above	5.82	8.30
(*c*) Pennsylvania citrate, rubbing thoroughly in a mortar with bare pestle	6.00	8.28
Insoluble:		
(*a*) As above	7.47	6.97
(*b*) As above	7.41 7.32	6.97
(*c*) As above	4.19 4.21	6.42

These tests were made a few weeks after those upon the unground samples. The two solutions of citrate were very similar in approach to neutrality, and in specific gravity.

Insoluble P_2O_5, by various analysts.

	A.	B.
	Per cent.	*Per cent.*
Chazal	3.90	5.73
Gascoyne	5.08	6.33
Gaines	7.84	8.64
Richardson	9.28	7.42
Jenkins:		
Using Pennsylvania citrate	9.34 9.61	....
Using Connecticut citrate	10.43 10.85	....

Jenkins states that the Pennsylvania citrate was somewhat more acid than that made at the Connecticut Station, which had been freshly prepared.

These results, carefully made, as they were, by different chemists, seemed to point distinctly to the fact that as to dissolved bone, at least, the association had not arrived at a satisfactory stage of progress, and also showed that in regard to many details of manipulation the chemists differed considerably. He thought the difference was principally due to various methods of minor manipulation, and would be glad to have some suggestions made which should lead to greater uniformity in the handling of bone goods, and wished that the reporter on phosphoric acid would send out samples of dissolved bone next year to be wrestled with. Results with ground rock agreed very exactly, but required careful handling for good results.

Mr. Gaines said that sample No. 3 was a mixture of dissolved bone and Charleston rock, or rather a mixture of dissolved tankage, which was the same thing, as the phosphoric acid in it was from the same source as that in bone; it was bone itself, and the greatest variation in that case, with fifteen chemists, was .44 of 1 per cent., though some of the chemists, he judged, were unaccustomed to doing very much fertilizer work. In the instances brought before the association by Mr. Frear the variations from an average would be something like 2 or 3 per cent. He believed that the London Board of Trade, two years ago, in arranging a scale by which the differences between chemists might be settled, agreed that a difference of .75 of 1 per cent. was near enough in the examination of phosphoric acid, while here the greatest variation in the determination of insoluble phosphoric acid in the samples sent out was one-half of 1 per cent.

Mr. Frear said that he should have stated before reading his paper that with two exceptions the samples were taken from the same jar at exactly the same time, and were very carefully mixed, especially after the first results; they were sent off in sealed tubes, and it could not be claimed that the men who analyzed them were incapable.

Mr. Scovell said that he would like to ask whether the samples sent out by Mr. Frear were large samples. In bone fertilizers an average could not be obtained from a small sample. He had visited factories where bone-meal was kept running from a spout, and one could pick up four or five shovelfuls and get different results. He had even tried mixing it in a pint bottle, and had found that unless it were cut in two accurately it would not give the same proportion. He had received from one or two persons specimens which he would not guarantee, their quantity was so very small.

Mr. Battle said that, with one or two exceptions, the samples were ground in his laboratory, so that difference in the respect referred to was eliminated as far as possible. The sample was small because he had but little material to work upon.

Mr. Gaines asked whether the samples sent to the chemist by Mr. Frear were portions of one original sample or of different samples, drawn at different times.

Mr. Frear said that they were all drawn at the same time.

Mr. Gaines said that as to the two samples sent him there were about 6 or 8 grams in each case, and they were rather coarse. He had had to compare them before he could begin the analysis.

Mr. Battle said that the material he referred to had been put through a 20-mesh sieve. He would not grind it finer than that, and felt free to say that he did not think it ought to be ground very fine.

Mr Voorhees said that in the case of bones much depended upon the manipulation after getting the treated sample to the filter. He had had considerable experience with material containing bone, and had noticed that whenever the filtrations were allowed to run beyond a certain time the determinations would not agree. For example, two determinations from the same sample, filtered for the same time, would probably agree in results, but if the filtering in one case was for half an hour, and in the other for fifteen minutes, the determinations would vary from three-tenths up to one-half per cent., and perhaps more. He thought it necessary to be very careful in the manipulation of the insoluble acid after it came from the bath, and it had been his custom to use a pump, but he had been unable to filter with sufficient rapidity to get results that would agree. By filtering through folded paper, however, the filtrations were more rapid and the results more uniform. He ground all samples fine enough to pass through a 25-mesh sieve, and with present methods found no serious difficulty in getting uniform results upon insoluble determinations with samples containing large amounts of bone.

Mr. Jenkins said that the samples he had received from Mr. Frear had seemed to him altogether too coarse. He did not remember whether he had ground them before making the determinations or had let them go as they were, on the supposition that the determinations were desired on the material just as it was, but he did recollect saying, when he looked at them, that they were altogether too coarse. Duplicate determinations that would agree very well could be made from a pretty coarse sample, provided it were thoroughly mixed. With regard to the grinding, he felt no hesitation in grinding everything, and in his State fertilizer control work did grind everything that he analyzed, to pass through meshes the twenty-fifth of an inch in diameter. That was what he called an average sample. If it was not in that condition when he received it from the manufacturer, he made it so; then any one gram of it would represent five hundred grams or a ton, of the material, but this could not be unless the sample would pass through a one-twenty-fifth-inch-mesh sieve. He thought that unless there was great danger of losing moisture, it was well to follow the plan suggested by the New Jersey Station, mixing the material rapidly on paper each time before weighing out a sample for analysis. Bone, he believed, was very prone to separate if very slightly jarred, being of different grades of fineness.

The president said that the particular question before the convention was as to the use of the sieve—whether the fertilizers should be sifted or not—the present discussion having arisen from the reading of Mr. Frear's paper. While speaking to the question it should be remembered that the more general subject before the association was as to whether the fertilizer should or should not be pulverized and sifted. If the association would permit him to make a few remarks upon this topic, he would state that in the case of bone meal the quantity of available phosphoric acid shown would depend entirely, according to his own experience, upon the fineness of the pulverization. When he was State chemist of Mississippi, the chemists there, in analyzing bone meal, had considerable trouble with a man in New Orleans who every fall sent up a sample can of bone meal, containing ground horns, hoofs, back-bones, ribs, and sometimes the hard bones from the legs of the animals, ranging in size from such as would pass through a 60-mesh sieve down to such as would not pass through a 16-mesh sieve. When an analyst started in to the work, it never could be told where he was going to come out; he might get good, concordant results, but the probability was that he would never accord with anybody else, and in order to protect themselves the chemists adopted the plan of putting everything of that kind through at least a 40-mesh sieve, by which means system and comparatively good results were brought forth from otherwise interminable confusion. The habit of analyzing a fertilizer just as it came from the factory would inevitably get anybody who tried it into trouble; at least that had been his observation. If results were wanted that one could compare with others of his own and with those of his brother chemists, the fertilizer should be put through at least a 40-mesh sieve before the determination was begun, and should be well shaken, and the analyst should dip deep in taking out his sample, not using the outside. Samples should not be sent out in one ounce vials, but good sized samples should be furnished, so that the chemists may have a chance to mix for themselves. The shaking up in transportation would put the big pieces on top and the small ones on the bottom. He thought it rather a good thing that Mr. Frear was having some trouble; it was right that such an excellent chemist should occasionally get into difficulty; it would force him to learn something about the troubles had elsewhere than in his own laboratory. The speaker had been through the mill, and knew exactly what it meant, and what it was to be found fault with and dragged into court on just that kind of work. He was confident that if the chemists could not get their samples well mixed, the results obtained would not be reliable; this was especially difficult in the case of bone meal. He thought that the association should prescribe some degree of fineness to which the fertilizer should be reduced before analysis.

Mr. Gaines said that he thought it gratifying that the chemists who had worked on these samples this year had agreed so closely. He had been mistaken in his previous statement as to the differences. The widest variation between two chemists—the lowest and the highest—was .63 of a per cent., and the widest variation from an average was .3 of a per cent. on phosphoric acid after the preparation and sending out of the sixteen samples. He thought that showed the method to be very good; one of the samples had contained dissolved bone.

Mr. Wiley, on behalf of the executive committee, announced that it had been decided to invite the members of the association to meet together at a banquet arranged for the following evening; it was hoped that all would be able to attend.

On motion of Mr. Wiley, the meeting (5.15 p. m.) was adjourned.

SECOND DAY.

MORNING SESSION, WEDNESDAY.

The president, Mr. John A. Myers, called the meeting to order at 9.30 a. m., and stated that the subject under consideration was the specification of a sieve to be used in the mixing of the samples under the head of phosphoric acid, a 20-mesh sieve being suggested.

Mr. Jenkins moved to amend by substituting a sieve with circular holes one-twenty-fifth of an inch in diameter.

Mr. Caldwell accepted the amendment, and the motion was carried.

Mr. Frear stated that the method at present used required a washing and rubbing in a beaker with a rubber-tipped pestle to a homogeneous paste. In his laboratory Mr. McDonnell had this year made some very careful comparisons between determinations of soluble and insoluble made by this method, and also by the method employed some time ago, which was simply washing the material upon the filter. He had been somewhat surprised at the results obtained; the rubbing had been moderate, only sufficient to avoid grinding, especially in the case of dissolved bones. The mean difference, taking the twenty cases, was .06 per cent. in favor, in the case of the soluble, of that washed directly upon the filter. In one case where the percentage of soluble ran up to 8 or 9 per cent., the results obtained by the rubbing and decanting from the beaker were 8.68 as compared with 9.48 of soluble obtained by washing directly upon the filter, showing that there must have been, in spite of the care to use only a small quantity of water and to pour off completely before adding fresh water, a precipitation in the beaker. In that case the results were duplicated because they were so far apart, and the results obtained by rubbing in the beaker were 8.64 and 8.72 per cent.; those by washing on the filter 9.52 and 9.44 per cent. The insolubles in the same samples were determined with a mean difference of less than .01 per cent.; a maximum difference of about .4 per cent.; in favor in that case of rubbing in the beaker, and in this last case where there was a difference in favor of the rubbing the difference in soluble was .15 per cent. in favor of washing on the filter. The length of time required was, according to his experience, somewhat greater by the rubbing method, and the danger in case of samples of dissolved bone was, especially in the hands of inexperienced chemists and trade chemists, that they would be rubbed too far. He found differences of .5 per cent. and more in his own hands where he rubbed very hard to get something approximating a homogeneous paste, but he never got it unless the samples were ground very fine. He would like to return to the method of washing directly on the filter, and made a motion to that effect.

The motion was seconded.

Mr. Harrington said that he was not now particularly interested in fertilizer analyses, but formerly had been, and had determined all his samples in the way preferred by Mr. Frear, which he thought was more accurate than the other, in which way there was not only the uncertain factor of rubbing up to an uncertain degree, but also a chance for loss.

Mr. McDonnell said that he thought it would be very desirable to make the change suggested, for it did not seem to him possible to rub the sample in a beaker to a homogeneous paste, if he understood that term. He thought it necessary to grind the material in a mortar to an impalpable powder in order to make a homogeneous paste.

The president stated that the method now in use was as follows:

Water-soluble phosphoric acid.—Weigh out 2 grams in a small beaker, wash by decantation four or five times with not more than from 10 to 15 cubic centimeters of water, then rub it up in the beaker with a rubber-tipped pestle to a homogeneous paste, and then wash four or five times by decantation with from 10 to 15 cubic centimeters of water. Transfer the residue to a 9 cubic centimeter, No. 589 Schleicher and Schüll filter, and wash with water until the filtrate measures not less than 250 cubic centimeters. Mix the washings. Take an aliquot part (corresponding to one-half gram) and determine phosphoric acid, as under total phosphoric acid.

The amendment proposed to strike out and insert so that the paragraph should read as follows:

> *Water-soluble phosphoric acid.*—Weigh out 2 grams, bring on to a 9-cubic centimeter, No. 589 Schleicher and Schüll filter, and wash with water until filtrate measures not less than 250 cubic centimeters.

Mr. Battle said that the change proposed seemed to him a very radical one. The association had never yet done away with the grinding in the mortar or the beaker, and he was not certain about the advisability of changing the method now. If it was desired to do so at some future time, he thought that the method adopted in regard to the incineration should be adopted in this case; that is, the reporter should be advised to suggest that the new method be tried on the samples sent out and should report on the same next year.

Mr. Jenkins said that last year when the change from the method formerly employed was made he was not present and did not know what experiments were adduced showing the necessity of such change, which had been a surprise to him; it had caused considerable grumbling in the laboratory, the new method being complicated and involving more chance of loss of soluble phosphoric acid than the other method. As he remembered, the method employed previous to last year was the same as that now proposed, with the exception that after washing the phosphate on the filter with small portions of water the residue was washed from the filter into a mortar and rubbed gently with a rubber pestle made particularly for that purpose. The idea was this, that if considerable water were thrown on a phosphate containing iron or alumina there was at first a solution of phosphates soluble in a concentrated-solution, but when this was diluted these phosphates were thrown down. In other words, certain phosphates soluble in a strong solution of phosphoric acid or soluble phosphates become insoluble upon dilution. The idea of the association had been that when only a small quantity of water was put on the filter, at first the iron and alumina phosphates were carried through, while a large quantity of water would make them insoluble again and leave them on the filter. Such a precipitate was found sometimes at the apex of the filter, which enveloped or might envelop small quantities of the phosphate, preventing further washing by water. To obviate this, after most of the phosphoric acid was washed out, the funnel was rinsed into the mortar and the substance was rubbed up with a rubber-tipped pestle so gently as not to break down the hard lumps, but only to crush up that smear of precipitated phosphates on the sample, and then was washed back on to the filter, where the washing was completed. He preferred a return to that method, which had been employed for years and which the association had been led to adopt by the results of investigation.

Mr. Scovell said that he agreed with Mr. Battle that it would be a radical change to abandon the rubbing with the pestle. He had had to do with a certain line of fertilizers, probably not used in Pennsylvania, and his results had differed as high as a half per cent., and in fact were always a little low in soluble or available phosphoric acid; in some cases he thought the difference would amount to fully 1 per cent., but he thought the rubbing with the pestles should be continued, although without it the results might be more even; he believed it fair to the manufacturer as well as to all others concerned that the total soluble or available phosphoric acid should be gotten out if possible.

Mr. Jenkins said that while he was willing to go back to the method of the year before last, he still desired the change that called for rubbing with a pestle; he thought that, so far as practicable, all the phosphoric acid should be gotten out. He would rather vote for the resolution for rubbing with a pestle.

Mr. Frear said that he was perfectly willing to modify the motion so as to include a slight rubbing with the pestle, but disliked the decanation into the beaker and rubbing as now practiced. He would like to see a large series of samples tested by the different analysts next year by washing directly upon the filter, as compared with washing in the beaker.

He would modify his motion so as to include the transfer of the material on the filter to a mortar, rubbing with a rubber-tipped pestle, and returning to the filter and making up just the same as it was year before last; he believed that was exactly the form; he thought it would be far safer than the present method and much less troublesome.

Mr. Battle asked whether the change proposed was merely a going back to the old method.

The president stated that it was proposed to return to the scheme of 1887–'88.

Mr. Frear said that he wished to urge upon the analysts who had a good deal of this work to do, that they should make a test of the method he had mentioned, washing directly on the filter without any rubbing whatever, and transferring directly to the flask by washing with ammonium citrate. This test could be made without great expense or trouble.

Mr. Caldwell said that in his opinion the association ought to be very careful in regard to changes of methods; nothing was more likely to affect the weight of the decisions rendered by the association than the making of changes without careful consideration and test. Although it seemed to him best to return to the method of 1887, he thought it must be confessed that to do so would add nothing to the authority of the association. A change had been made in 1887, another in 1888, and now in 1889 it was proposed to go back to the method of 1887, and this without any very careful tests.

Mr. Gaines said that the tabulated results for soluble phosphoric acid on the samples sent out agreed quite closely where the analyses were made during the second week in January, as was requested. Some of the analyses were not made until late in the spring, and still others not until summer, and those were lower in soluble phosphoric acid. It seemed to him, however, that the proposed change was not shown to be necessary by the results obtained on the samples. According to an examination that he had made, the agreements with the figures of previous years in each determination of phosphoric acid as soluble and as insoluble had been closer than ever before, which would indicate that the association was nearer to a true method.

Mr. Terne said that as a chemist and manufacturer he felt that the more closely the association adhered to methods which had given entire satisfaction the better it would be for its authority in assuming to direct the analytical work of the country. If changes were made from year to year, the work in the outside laboratories would be upset unless such changes were founded upon experiments made and results presented. He thought that in making alterations great care should be exercised.

Mr. McDonnell said that he would rather see the method remain as it was than to return to the old plan. It was certainly less simple to transfer the substance from a filter to a mortar and back again. He did not believe in continual rubbing and sifting, but would rather see the method at present employed continued than changed to the old plan. If the substance were properly sifted and stirred each time and water added in extracting the soluble, it was unnecessary to rub it. A rubber-tipped pestle could only be used to stir; it could not grind.

Mr. Scovell said that he regretted the absence of Mr. Gascoyne, who had moved the method adopted last year and who had made a number of experiments showing that the results of the two methods agreed closely, an overwhelming number of examples having been presented.

Mr. Frear said that he desired to have all the results obtainable, and if Mr. Gascoyne had notes that would throw more light upon the matter he should be glad to have the motion laid on the table until Mr. Gascoyne should arrive.

It was moved that the subject be laid upon the table. The motion was seconded and carried.

Mr. McDonnell said that in making ammonium citrate solution he saw no reason why the hydrate should not be used. It was certainly cheaper and a large quantity could be made much quicker and easier than by the use of the carbonate.

The alternate method for potash might be shortened by omitting filtration after the addition of barium chloride and hydrate; ammonium hydrate and oxalate are also omitted.

The president asked whether Mr. McDonnell made a motion to that effect.

Mr. McDonnell said that he did not think he was entitled to a vote.

Mr. Jenkins said that as he understood it, ammonium citrate could be made up in any way preferred. The directions were, he believed, to heat to expel carbonic acid, and so long as the carbonic acid was expelled he did not see that the method was of account. It was necessary to be assured that pure ammonium citrate was obtained, and that was all.

Mr. Burney said that while it was only necessary to get ammonium citrate 1.09 it seemed to him desirable, in the light of the experience he had had, that the association should adopt some one indicator for the purpose of determining when the solution was neutral. He found it a matter of no small difficulty to determine just when the ammonium citrate solution was neutral. Indeed, the use of litmus paper and of coralline would give results not concordant. The coralline said that the solution was acid, when the litmus paper said that it was neutral. He should like to hear the experience of other members in regard to that matter. It seemed to him that coralline should be used as the indicator for determining the point of neutrality in making up that solution. He had always used the coralline, but supposed the litmus was also allowed.

The president stated that coralline was prescribed by the regulations, which read, "Add ammonia until exactly neutral (testing by saturated alcoholic solution of coralline)."

Mr. Battle said that he wished to inquire, in reference to the water soluble phosphoric acid, the amount of water required to wash completely. In his laboratory he had always found 250 cubic centimeters too small an amount, and had invariably washed to 400. He did not know what had been the experience in other States; if it had been similar to his own, he thought that 250 should be changed to 400, and should like to hear from some of the members present upon that point.

Mr. Frear said that he had made a few tests, but could not give the exact results. He found that in some cases by the time 250 cubic centimeters washings were put through, there was still a very faint acidity in the drops coming through. He then took 250 cubic centimeters more and determined the amount of difference there would be by taking the aliquot and determining the phosphoric acid, and found the additional phosphoric acid was not sufficient to go beyond the limits of error in manipulation; so that for practical purposes 250 cubic centimeters appeared to answer in all the experiments he had made. He did not recollect the specific instances, but remembered the fact.

Mr. Battle said that in North Carolina $3,000,000 worth of fertilizers were sold every year, and owing to the magnitude of the trade he thought that as much time as possible should be given to the matters which gave trouble and caused differences in fertilizer analyses. In regard to the citrate insoluble, the method stated that after the flask was put in the bath, the temperature was to be raised as rapidly as possible to 65° C., which temperature was subsequently maintained, and the further direction was to "digest with frequent shakings." He would like to know what was meant by the words "frequent shakings."

The president said that he supposed it means that the bottle was to be shaken.

Mr. Battle said that that was where the trouble came in. How many times should it be shaken during half an hour? If shaken every two minutes, there would be fifteen shakings during the thirty minutes. If shaken every five minutes, there would be six; and if shaken every ten minutes, there would be three; but it was clear that upon the number of shakings would depend the decrease or increase of insoluble phosphoric acid. He had always shaken every five minutes, six times during the half hour. He thought that some number of shakings during the thirty-minute period ought to be specified and prescribed, and moved that in the method, the words "digest with frequent shakings for thirty minutes" be substituted by the words "digest for thirty minutes, shaking every five minutes."

The motion was seconded and carried.

Mr. Jenkins moved that the motion previously laid upon the table, to await the arrival of Mr. Gascoyne, be taken up and further discussed, Mr. Gascoyne being now present.

The motion was seconded and carried.

Mr. Gascoyne said that he believed the only reason for making the change in the process in 1887 was the supposition that the method then proposed would prevent precipitation of aluminic and ferric phosphates in the filter. It had been found that in washing the filter some phosphate would be precipitated and formed in the bottom of the filter, and his own experiments had proved that higher results were obtained by the method adopted, which, he believed, was the same method used by Mr. Chazal, of South Carolina. He knew of no other reason for the change made. He would like to ask the reporter how the results agreed this year.

Mr. Gaines said that they agreed very well, better than at any previous time, so far as he could judge from such cases as he had had of the publications of the meetings of the association. He thought that the greatest variation from an average was something like .4 per cent., but where the analyses were made at the time requested (the first week in January) the agreements in soluble phosphoric acid were very close. Those chemists who had analyzed their samples late in the summer got lower results.

The president stated that he should see with a great deal of reluctance any changes made without somewhat serious grounds, and he hoped that the convention would consider very carefully any alterations made in the regular processes already adopted. He was disposed to agree with Mr. Caldwell that it was bad policy to vacillate back and forth on these methods. He would put the question unless some one else had further remarks to make in regard to the subject.

Mr. Gascoyne said that the method now in use seemed to him to have given tolerably uniform results. He thought that the report of last year from which he quoted bore him out in this belief.

The motion was lost.

Mr. Frear said that he would like to have the reporter on phosphoric acid for next year requested to include in his scheme a test of the direct washing upon the filter without rubbing, in order to see what difference really existed between the results obtained in that way and those obtained by decantation; some of the results by the latter process had shown 1 per cent. in favor of washing on the filter.

Mr. Gascoyne asked whether the results obtained by washing on the filter were higher.

Mr. Frear said that they were higher by over 1 per cent.

Mr. Gascoyne asked whether Mr. Frear had used a large amount of water.

Mr. Frear replied that he had used 250 cubic centimeters.

Mr. Gascoyne said that by using a large amount of water a dilute solution of phosphoric acid would of course be made.

Mr. Frear said that that had been carefully avoided.

Mr. Gascoyne said that it was often noticeable in a filtrate that after standing for some time it became cloudy by the separation of the iron and alumina phosphates.

Mr. Frear said that in the course of his investigations on the bone samples he had tried to find out whether there was any difference in the manipulation practiced by the different chemists in the process of extracting the "revert," so called, in dissolved bone, and he had found that the greatest point of difference was in the method of filtration and washing the precipitate. In some cases, the filter pump was used, requiring a great amount of time, while in others the filter alone was used; in some cases wash-water seemed to be used before the citrate solution had run through the filter; and in one case he had been unable to get the chemist to say whether he had allowed these several portions of wash water to run through separately before putting on a fresh portion. He simply called attention to these results because the experience ought to convince some of the members of the necessity of a closer agreement in little details of manipulation on some kinds of goods.

Mr. Gascoyne said that he was the chemist alluded to who did not reply to Mr. Frear's letter. He would say now that he always allowed the citrate to go through first and then washed with water from three to five times. In the case of the particular sample of bone referred to, it took, if he remembered right, about two hours to complete the filtration. There was a good deal of fatty matter in the bone that seemed to clog up the pores of the filter, and it had been quite a task to get it through. That might account for some of the differences in the analyses, but he was inclined to think that the principal difference lay in the sample. His sample was not ground, and a large portion of it would not pass a 20-mesh sieve; some of it would not go through a 40-mesh sieve. He thought it hardly possible to get a fair test out of such a sample as he had, which was evidently a mixture of dissolved bone and raw bone, or bone very slightly acidulated.

Mr. Burney said that some filter pumps were good and others were not, and that in this fact there was a source of diversity of usage. If a uniform method could be adopted, say the use of fluted filters of 12½ centimeters diameter, and some one kind of paper, the difference in time of filtration in the several laboratories would not be appreciable. If one chemist used a good filter pump, another an inferior one, and a third used the fluted filter, the length of time during which the substance was exposed to the action of the ammonium citrate would vary considerably, and it seemed to him that the results must necessarily vary under these differing conditions. He felt disposed to make a motion to the effect that a 12½-centimeter fluted filter be used, and that some special kind of filter paper be adopted for the purpose, but he would make such a motion with considerable hesitation, because he had had but one year's experience in this kind of work; he very much preferred that some one else should take the initiative in that matter.

Mr. Gascoyne said that he had had very little trouble with getting citrate of ammonia through; his trouble has always commenced with the washing. It seemed to him that the washing with cold water separated the fat. The citrate of ammonia went through quickly enough.

The president asked whether Mr. Gascoyne had had any cotton-seed meal.

Mr. Gascoyne said that he sometimes had it, but only in fertilizers. He knew that cotton-seed meal would stick.

It was moved that the scheme of analysis recommended by the reporter on phosphoric acid, as modified by the several amendments proposed and made, be adopted as a whole. The motion was seconded and carried.

The president called for the report of the committee on organization of a national chemical society.

Mr. Wiley said that he should occupy but a very few moments with his report, and would then ask that the courtesy of the association be extended to Mr. Clarke, who would speak on the same subject. They had taken action by correspondence, and the letter of Dr. Prescott, chairman of the committee of the chemical section of the American Association for the Advancement of Science, had been submitted to the committee of this association, whose views thereon were elicited. The committee were unanimously of the opinion that the organization of a national chemical society would prove of the greatest advantage to the chemical interests of the country. The reasons for this opinion might be briefly stated. At present, only a very few of the chemists of the country derived any advantage from any chemical organization. The only chemical organizations existing in the United States were: (1) the Chemical Section of the American Association for the Advancement of Science; (2) the American Chemical Society, located in the city of New York; (3) the Chemical Section of the Franklin Institute of Philadelphia; (4) the Chemical Society of Washington; and to these might be added (5) a part of the organization of the Institute of Mining and Civil Engineers; they had a kind of chemical section, or at least discussed frequently chemical subjects.

Aside from those named and this association, no other chemical organization was in active operation in any part of the country. Other chemical societies had existed, but those were now defunct. For the purpose of affording chemists in all branches of the profession an opportunity for working together, he advised the organization of a national chemical society. It had been found, however, that in some quarters of the country there was opposition to the organization of such a society. One difficulty was the present existence of the society claiming to be the American Chemical Society; but the latter had become a purely local society, and was, he might say, confined almost exclusively to the city of New York. He had once been a member of that society, and supposed that many of those present had been and perhaps might still be members; but the sole benefit which he, at that time located in a distant part of the country, received from the American Chemical Society was simply a receipt for his annual dues. He had therefore, after paying his dues, resigned. It was now time that in the organization of a national chemical society the chemists of the country might be brought together in an association strictly and purely American in its character. It was intended not to confine it to the United States, but to include the chemists of Mexico and Canada, and in fact the whole continent of North America. This was not the time and place for outlining the opinions formed by the committee in regard to the method of organization, a matter which must be deferred until later; it was only meant to report the facts already mentioned and to urge upon the association of official agricultural chemists the continuance of a committee for the purpose of further considering this important subject.

The president extended the courtesy of the house to Mr. F. W. Clarke, and invited him to present his views in regard to the subject under discussion.

Mr. Clarke said that the movement in question originated a year ago in the Chemical Section of the American Association, at which time a committee was appointed consisting of Messrs. Prescott, Springer, and Hart, who were directed to report upon the matter. That committee, after conference with committees appointed by this association, the Washington Chemical Society, the American Chemical Society in New York, and the Chemical Section of the Franklin Institute, presented a report drawn up by Mr. Prescott, extremely moderate and judicious in tone, but favoring the idea of a national chemical society. The report was discussed to some extent, and a motion was made, which, being in some of its details unsatisfactory to certain members of the section, was lost. As a result, a new committee was appointed by the Chemical Section to report further progress next year. That committee was composed of the speaker as chairman, Messrs. Bolton, Hart, Warder, and Ellis. He [Mr. Clarke] had had a number of conversations with other chemists upon the subject, most recently with Messrs. Bolton and Langley. Mr. Langley thought it very important that the technical chemists of the country should be brought into rapport with each other. A large number of chemists were employed in manufacturing establishments, and many of them were doing admirable scientific work which was unknown for want of publication. American chemists could get more information about what was being done in Europe than about that which was going on in their own country. It was thought that the chemists in the American Institute of Mining Engineers were hardly more than tolerated and felt themselves to be mere hangers on, and most of them if not all would gladly welcome an organization which brought all the chemists of the country into union and had a journal in which all American work could be reported in systematic form. In the discussion at Toronto the main difficulty had appeared to depend on the possibility of printing a journal, and doubts had been expressed as to whether a journal could be carried on by the proposed society. The speaker thought that there would be no difficulty in securing a membership of 500, and as the Russian Chemical and Physical Society, having only 300 members, succeeded in publishing a very good journal, he did not think the outlook absolutely hopeless for the American chemists, although there were some people who were not inclined to believe that anything could be done on this side of the water.

The general consensus of opinion seemed to be that in order to avoid the danger of localizing the society it would be necessary to follow to some extent the example of the Society of Chemical Industry in Great Britain, which has a number of sections in different parts of the Kingdom. There was a London section, a Liverpool section, sections at Manchester, Birmingham, Newcastle, Glasgow, and other points, the several sections all co-operating through one journal. Probably the same plan would be feasible in this country, where there were already a goodly number of centers of chemical industry. It might be claimed that Washington, with an active society of forty-nine members, was such a center; Philadelphia was another; Baltimore, perhaps, might be counted; and New York, including Brooklyn, was one more. Boston and its surroundings should also be considered; in the Institute of Technology alone there were sixteen chemists engaged in teaching. Chicago had a good many chemists, commercial and technical; and quite a number also were to be found in St. Louis and Cincinnati. In the United States, according to the last census, there were 2,000. There were a considerable number scattered in different places who could not attend an annual meeting at a place remote from their homes, but who could attend meetings of sections held within their reach; men in Missouri might attend at St. Louis; men in New Hampshire could go to Boston; and chemists in the Gulf States could perhaps meet at Atlanta.

By such a system every member of the society could be within comparatively easy reach of some one section, while at the same time all would be likely to get the worth of their subscription fees in a journal which should be a common medium for all these sections. The policy of the journal might be shaped after that of the German Chemical Society; if one *subscribed* to the Berichte der Deutschen Chemischen Gesellschaft he paid 36 marks a year, while 30 marks would make him a member of the society with the journal thrown in; that policy had given the Berichte a very large subscription list, and he thought that if a journal were published by a truly national (not a local) society in this country a subscription price a little higher than the dues would insure a respectable membership and a reasonably good number of active sections. Mr. Langley was very strongly of the opinion that the society was needed; Mr. Bolton would ascertain the sentiment of the New York chemists, and the speaker could say that he knew of two other New York men who were in favor of it, namely, Mr. C. F. Chandler and T. Sterry Hunt, who had been presidents of the American (New York) Chemical Society. He thought that by another year the committee would be able to tell exactly the sentiment of the American chemists in regard to the matter.

The president extended to Mr. Clarke the thanks of the association for his remarks.

Mr. Caldwell moved that the report of the Committee on the Organization of a National Chemical Society, as presented by Mr. Wiley, be accepted and that the committee be continued. The motion was seconded and carried.

Mr. Bowman moved that the Association approve the proposition to organize a National Chemical Society. The motion was seconded.

Mr. Jenkins said that he should certainly be in favor of the motion if he were correct in understanding the approval expressed to be general in its nature, and not an approval of any specific scheme. He should be strongly opposed to any scheme which rendered it necessary for the Association to advise with other sections or with other societies as to the time and place of holding its meetings or which should oblige the Association to change the time and place at any disadvantage to itself for the convenience of others, or which should so change the method of publishing the results of the Association's work and its discussions in convention as to present them in a more abridged form than that in which they are now presented. He supposed, however, that no such changes were in contemplation.

Mr. Caldwell said that the action taken at Toronto accorded with that which seemed likely to be taken in this convention. At Toronto he had not been quite certain that there would be sufficient enthusiasm on the part of the chemists to justify the formation of the proposed society. If such a society could be organized and could receive hearty co-operation, all would be well, and the organization could certainly be effected. He thought that it could be done, and his only question was whether it was not somewhat premature to say anything about it at this time. The committee was continued, and a new committee had been appointed on the part of the Chemical Association. In his mind it was somewhat doubtful whether anything more should be said than sufficed to express, as Mr. Jenkins had suggested, a general approval of the idea without involving the approval of any feature of the scheme.

Mr. Frear said that he was warmly interested in the matter. He thought that the society would be of personal benefit to himself, for he very frequently needed access to information upon just such points as a journal of the kind proposed would probably contain. In regard to enthusiasm in the matter, he believed that if that were wanted by the association it would be a good thing for it to show its own. He had found that enthusiam was rather contagious, and that people sometimes became enthusiastic because they saw that others were so. If the members of the association believed that the matter in hand ought to be pushed, they should bring forward their enthusiasm and push it. He did not believe in being too far ahead of one's age or in showing it if one were so; that was sometimes very uncomfortable, but considering the number of chemists in the country, and the widespread interest which had been exhibited to himself in conversation with men of various sections, it did not seem to him that the movement was premature, and he should be very glad indeed to have the members present, coming as they do from all parts of the country, express by vote their approval of the general scheme for the formation of a national society having in view the general objects stated, without expressing an opinion as to the exact form of organization.

The motion was carried.

The president said that Mr. Atwater's remarks on the lines of investigation for chemists would now be in order.

Mr. Atwater said that he was aware of the extreme importance of the deliberations of the association, and that the time was short. He would endeavor to outline in the briefest possible form the ideas he had to present, and for which, personally, he wished there were more time. What he wished to urge especially was the need of more thorough study of the proximate compounds in animal and vegetable products, their molecular constitution and their potential energy. A more thorough knowledge of these things is needed for improvement of methods of analysis, and determination of digestibility, and of full value of foods and feeding stuffs. Many of the members were aware of the fact that in connection with the experiment stations of the country the Department of Agriculture, through its Office of Experiment Stations, was endeavoring to learn what were the ways in which it might be useful to the experiment stations, and what were some of the general lines of inquiry which might be aided by co operation and by the getting together of general information. In the prosecution of his own duties in that direction a number of considerations had arisen, the importance of which grew upon him as he found it had upon many others. The discussions of the association upon the methods of analysis emphasized one of those considerations, namely: that of a more thorough knowledge of the chemical constitution of the vegetable and animal products which were used for foods and feeding stuffs. What was the fundamental difficulty in the methods of analysis? Was it not more than anything else that the chemists did not understand the things with which they dealt? If the actual nature, constitution, and solubilities of the constituents of hay, of corn meal, of bran, of cotton-seed meal, of other products that the chemists had to analyze, were known, as they were not known at present, could not the chemists manage a great deal better with their methods of analysis? In short, was not a much more thorough understanding of the chemical constituents of the things dealt with needed? But there was another subject of no less importance, one which the methods of analysis were really meant to learn about; he referred to the nutritive values. One of a series of publications soon to be issued by the Office of Experiment Stations had to do with this general subject of the composition and nutritive values of feeding stuffs. As a beginning in that direction, Mr. Jenkins had undertaken a compilation of the analyses thus far made of these substances; Mr. Jordan was undertaking a compilation of what was known and especially what had been gathered in this country in regard to their digestibility. The speaker had frequently conversed with Mr. Wiley and other gentlemen in regard to a compilation of what was known, and further investigation as well. Those who had looked into the subject the most he thought were the ones most impressed with the importance of that kind of study. All were decided in their expressions regarding the unsatisfactory condition of our knowledge and the consequent unsatisfactory results obtained, and they believed that more thorough chemical study was what was necessary. He now found himself driven by the logic of events to the conclusion that the Office of Experiment Stations must make this matter of which he was speaking one of the important subjects for its study, and that all that could be done to secure the efforts of chemists on this subject was demanded by the very great, he might say enormous, interests of the feeding of domestic animals, to say nothing of the higher importance of the subject in connection with the food and nutrition of men. If there were time he should like to enlarge upon the need of study of the approximate compounds in animal and vegetable products, their molecular constitutions, and give illustrations, but his time was not long. This, it would be seen, was directly connected with the general subject upon which Mr. Caldwell had reported. He had felt that in doing the work which he was called upon to do he should be greatly assisted if he could feel that he had the co-operation of the gentlemen of the Association of Chemists, but perhaps if that were putting the matter in too much of a personal form, that the same principle which had already been adopted with so very great success and usefulness by this association could be carried further; and that the experiment stations would be very materially aided by the continuation of lines of work in the general direction exemplified in the reports already named. What he had especially in mind, however, was something a little different from that which Mr. Caldwell had reported upon, not simply a study of the methods of analysis, but rather a more general chemical study of our vegetable and animal products. He had not anything specific to ask of the association. What, it seemed to him, would be eminently useful would be simply the co-operation of the association as a whole, and especially of such of its members as were able to devote attention to the subject in the consideration, first of all, of plans for the more thorough chemical study of vegetable and animal products used for foods and for feeding stuffs.

It seemed to him it would not be inappropriate to mention the subject at this meeting and to ask for any action which the association might consider advisable in consideration on the one hand of the subject itself, and on the other hand of its own particular function, and of what was being done in the improvement of the methods of analysis by its committees. He should be very glad to hear any expression with regard to the matter before making any specific suggestions himself. Perhaps he might say one thing more. He had been very much interested in what had been said with regard to the methods of analysis and the references made to the experiment stations had of course interested him most of all. Whatever the Department could do through its Office of Experiment Stations to aid in the work of the association, for instance in such an enterprise as Mr. Caldwell had spoken of the day before, the getting of the chemists employed in the experiment stations of the country to co operate in studies of the methods of analysis, he should, as connected with the Office of Experiment Stations, be very glad to assist in. He would like to have the association assured that Mr. Wiley was not the only man in the department willing and glad to help the association.

Mr. Caldwell said that as for himself it was very possible he should be able to make use of the services so kindly offered by the Office of Experiment Stations, and he thought that the other reporters might, on thinking over their work and their plans for the year, make equal use of those services.

Mr. Richards read the following article:

THE WITHDRAWAL OF ALCOHOL FROM BOND FOR SCIENTIFIC PURPOSES, FREE OF TAX.

It may not be generally known to the members of this association that they can obtain their supplies of alcohol for use in their chemical laboratories free of the internal revenue tax of 90 cents per proof gallon, or $1.70 per gallon of 95 per cent. alcohol, by complying with certain regulations prescribed by the Secretary of the Treasury, in accordance with Section 3297 of the Revised Statutes of the United States.

THE LAW.

This section provides that "The Secretary of the Treasury is authorized to grant permits to any incorporated or chartered scientific institution or college of learning to withdraw alcohol in specified quantities from bond without payment of the internal-revenue tax on the same, or on the spirits from which the alcohol has been distilled, for the sole purpose of preserving specimens of anatomy, physiology, or natural history belonging to such institution, or for use in its chemical laboratory: *Provided*, That applications for permits shall be made by the president or curator of such institution, who shall file a bond for double the amount of the tax on the alcohol to be withdrawn, with two good and sufficient sureties, to be approved by the Commissioner of Internal Revenue, and conditioned that the whole quantity of alcohol so withdrawn from bond shall be used for the purposes above specified, and for no other, and that the said president or curator shall comply with such other requirements and regulations as the Secretary of the Treasury may prescribe. And if any alcohol so obtained is used by any officer, as aforesaid, of such institution for any purposes other than that above specified, then the said officer or sureties shall pay the tax on the whole amount of alcohol withdrawn from bond, together with a like amount as a penalty in addition thereto."

The above provisions are further extended by the act approved May 3, 1878 (20 U. S. Stat. 48), which provides:

"That the Secretary of the Treasury is authorized to grant permits, as provided for in section thirty-two hundred and ninety-seven of the Revised Statutes of the United States passed at the first session of the Forty-third Congress, to any scientific university, or college of learning created and constituted such by any State or Territory under its laws, though not incorporated or chartered, upon the same terms and subject to the same restrictions and penalties already provided by said section thirty-two hundred and ninety-seven: *Provided further*, That the bond required thereby may be executed by any officer of such university or college, or by any other person for it, and on its behalf, with two good and sufficient sureties, upon like conditions, and to be approved as by said section is provided."

DOCUMENTARY EVIDENCE REQUIRED FROM A SCIENTIFIC INSTITUTION BEFORE IT CAN ENJOY THIS PRIVILEGE.

To obtain this privilege for its chemical laboratory, an incorporated or chartered, or not incorporated or not chartered, scientific institution, university, or college of learning must, at the time of its original application, clearly show, by a copy of its charter, articles of incorporation, or other documentary evidence, that it is an institution duly entitled to such permit by possessing a suitably equipped chemical laboratory and otherwise.

FORM OF APPLICATION.

Its application is made by the president, curator, or duly authorized agent, "through the collector of internal revenue in whose district the institution for which the withdrawal is to be made is situated," "to the Secretary of the Treasury for permit" "to withdraw from the distillery bonded warehouse, owned by ———, at ———, in the ——— district of ———, the alcohol which was stored in said warehouse on the ——— day of ———, 18—, described as follows, viz, number of packages; marks and serial numbers of packages; numbers of warehouse-stamps; wine-gallons; degree of proof; proof-gallons; taxable gallons; and amount of tax; for the *sole purpose* of use in the chemical laboratory of the institution, located at ———, in the State of ———, of which institution" the signer is one of the persons above mentioned.

"The application in all cases must be sworn to, or affirmed, before an officer authorized to administer oaths."

CHARACTER OF THE BOND.

The bond, required to be filed with the first application, must be in a penal sum of "not less than $200, and never less than double the amount of tax on the alcohol withdrawn at any one time," is signed by the applicant "as principal, and with two or more sureties, who shall not be officers of the institution in which the alcohol is to be used, but shall be residents of the United States judicial district in which such institution is located." In the case of "an incorporated institution, its name should be signed to the bond as principal, and its corporate seal affixed by its duly authorized officer, who should also sign his own name as such officer."

The bond is conditioned that "the entire quantity of alcohol" intended to be withdrawn "from distillery warehouse, without payment of tax," by the said principal, will be "for the sole purpose of use in the chemical laboratory of the" said institution, "in the city or town of ———, of the county or parish of ———, and State of ———," "and for no other purpose."

The bond is known as a "continuing bond;" *i. e.*, "withdrawals may be made from time to time," "by the said principal," "until this bond shall have been revoked or canceled by direction of the Secretary of the Treasury," of certain alcohol for use "by the said institution, or the proper officer thereof, for the purpose above specified, and for no other purpose." "The principal of said institution shall, as to each lot of alcohol so withdrawn, produce within" the time "fixed by the collector accepting the bond," "from the date of such withdrawal," "proof satisfactory to" the collector of internal revenue of that district "and to the Commissioner of Internal Revenue that the said alcohol has been so used for the purpose above specified, and for no other purpose." He shall also "comply with such other requirements and regulations as the Secretary of the Treasury may prescribe;" and according to the last paragraph of section 3297, Revised Statutes, "the said officer or sureties" are bound by this bond to pay to the collector "the tax on the whole amount of alcohol withdrawn from bond, together with a like amount as a penalty in addition thereto" "in case said alcohol, or any part thereof, shall be used for any purpose, other than that specified."

BOND AND APPLICATION TO BE FILED WITH THE COLLECTOR FOR APPROVAL.

The "bond, together with the first application for permit, will be deposited by the applicant with the collector of internal revenue for the district where the institution in which the alcohol is to be used is located." It is the collector's duty to forward the bond and application to the Commissioner of Internal Revenue, with his certificate of approval, etc. He keeps "a copy of the bond, or a memorandum of its date, penal sum, and the names of the signers; and whenever an application for alcohol is made after the first one, the collector will certify to the Commissioner of Internal Revenue that the bond remains good, or will notify him of any change affecting the responsibility of the signers." "Upon the approval of the bond by the Commissioner of Internal Revenue," applications may be made on the prescribed form "for the withdrawal of alcohol as occasion may require" by the duly authorized officer or agent: "*Provided*, The penal sum of the bond is equal to double the amount of tax on the alcohol to be withdrawn, after deducting all outstanding charges on the bond." The Commissioner transmits the applications "to the Secretary of the Treasury, with a notification of the approval of the bond when the first application is forwarded, and afterwards with a reference to the bond under which the application is made."

CANCELLATION OF BOND.

To cancel the bond "or for the purpose of obtaining a credit on said bond" a sworn certificate is "required of the officer or officers of the institution under whose direction or supervision the alcohol has been used." The certificate is "filed with the collector named in the bond, and, if approved, to be forwarded by him to the Commissioner of Internal Revenue with his approval indorsed thereon."

EXTENSION OF TIME NAMED IN BOND.

It sometimes happens that the alcohol is not entirely used up in the time specified in the bond, and that consequently "the principal to the bond is unable, from good cause, to furnish the required proof" of such use. He may obtain an extension of the time named in the bond upon application to the Commissioner of Internal Revenue, "accompanied by the consent of the sureties to the bond to such extension; such application and consent to be approved by the collector with whom the bond was originally filed." "The extension asked for must be for a specified time," "not exceeding one-half the period named in the bond." The application "must be sworn to," and state "the reasons why the condition of the bond as to the presentation of proof has not been complied with." The consent of the sureties must be "under seal, and witnessed as in the case of giving an original bond."

PERMITS TO BE ISSUED BY THE SECRETARY OF THE TREASURY.

"The Secretary of the Treasury will issue," "upon receipt of the application" "and notice of the approval of the bond," "and transmit through the Commissioner of Internal Revenue, a permit in duplicate, one copy of which will be forwarded to the applicant and the other copy to the collector of internal revenue for the district in which the distillery warehouse is located," to withdraw from the specified warehouse the number of proof gallons of alcohol described in said application.

The collector notifies "the store-keeper at the bonded warehouse from which the spirits are to be withdrawn" of the receipt of the permit, a copy of which is sent to him, and authorizes him "to deliver the spirits to the person named therein, or his duly authorized agent, without the payment of tax, upon delivery to such store-keeper and cancellation by him of the duplicate permit issued to such person." This "cancellation shall be made by writing across the face of said duplicate permit the words 'The spirits herein described were delivered to the person herein named this ——— day of ———, 18—;' to be signed by the store-keeper." "There shall also be indorsed on the back of said permit the following receipt: 'Received the spirits within mentioned this ——— day of ———, 18—,' which receipt shall be signed by the person named in said permit."

Such in detail are the steps to be followed as prescribed by the regulations of the Secretary of the Treasury under date of March 26, 1889.

Blank forms are not furnished by the Treasury Department; and such forms, either printed or written, must be supplied by the parties making the application and bond. The forms to be followed are contained in Circular No. 34, 1889, of March 26, 1889, Treasury Department.

ARE AGRICULTURAL EXPERIMENT STATIONS ENTITLED TO THIS PRIVILEGE?

The laws quoted above restrict this privilege of the withdrawal of alcohol free of tax to four specified beneficiaries, viz: (1) "any scientific institution" or (2) "college of learning," "incorporated or chartered;" (3) "any scientific university," or (4) "college of learning," "created and constituted such by any State or Territory under its laws, though not incorporated or chartered;" which are further qualified as using alcohol (*a*) to preserve specimens of anatomy, (*b*) physiology, or (*c*) natural history, or (*d*) to be employed in its chemical laboratory. With the latter qualification the members of this Association are chiefly concerned.

The act, approved March 2, 1887, "to establish agricultural experiment stations in connection with the colleges established in the several States under the provisions of an act approved July 2, 1862, and of the acts supplementary thereto" (24 U. S. Stat., 440), commonly called the "Hatch bill," provides in section 8 that these "agricultural experiment stations established by law" may or may not be "in connection with any university, college, or institution not distinctly an agricultural college or school," or may or may not be "separate from" "colleges entitled" "to the benefits of this act;" *i. e.*, land-grant agricultural colleges.

Section 2 provides "that it shall be the object and duty of said experiment stations to conduct original researches or verify experiments on * * * the chemical composition of useful plants at their different stages of growth; * * * the analysis of soils and water; the chemical composition of manures, natural or artificial; with experiments designated to test their comparative effects on crops of different kinds; * * * the value of grasses and forage plants; the composition and digestibility of the different kinds of food for domestic animals; the scientific * * * questions involved in the production of butter and cheese; and such other researches or experiments bearing directly on the agricultural industry of the United States as may in each case be deemed advisable, having due regard to the varying conditions and needs of the respective States or Territories."

These experiments evidently require a well-equipped chemical laboratory in which alcohol would be often used as a solvent and re-agent in addition to any use of it as a fuel in the laboratory.

Taking the above quoted qualifications and provisions of law into consideration, it would seem that any agricultural experiment station that is established in conformity to the provisions of the act of March 2, 1887, by any State or Territory as such, when such station is connected with a "university or college of learning," is entitled on presentation, by its duly authorized officer or agent, of documentary evidence to that effect submitted to the Secretary of the Treasury through the Commissioner of Internal Revenue and the collector of internal revenue of the district in which the experiment station is situated, and by filing a bond and otherwise conforming to the regulations above specified, to the privilege of withdrawing alcohol free of tax for use in its chemical laboratory; also that when the experiment station is not connected with a college, etc., it may obtain free alcohol under the same regulations, provided it is shown to the satisfaction of the internal revenue officers and the Secretary of the Treasury that it is of itself a "scientific institution" as construed by those officers, that is, such an institution whose object is educational.

EDGAR RICHARDS.

The president said that the matter of securing alcohol for the stations was one of considerable importance; he trusted that Mr. Richards's paper would be incorporated in the proceedings. It would be found that it took some time to get the alcohol through. Papers of his own had now been in the Treasury Department for three months; it usually took about six.

Mr. Jenkins, referring to the remarks of Mr. Atwater, moved that a committee be appointed to consider the report upon plans for more thorough chemical study of foods and feeding-stuffs, with the purpose of obtaining such information as should aid in the improvement of methods of analysis and in the more satisfactory estimation of nutritive values.

The motion was seconded.

Mr. Jenkins said that in connection with the motion he would state that while the direct object of the association was not the scientific investigation of the nature of foods and feeding-stuffs, it still seemed to him that a certain amount of such information was made proper for the Association to undertake by reason of the fact that the chemical analyses of such subjects were interesting to it, and these analyses should be governed by the chemical nature and structure of the bodies dealt with.

It seemed to him, therefore, that the motion was a proper one. He should be very glad indeed to have a committee appointed, for a more thorough chemical knowledge of the proximate ingredients of our animal and vegetable products was now a crying need.

Mr. Atwater said that it had not been his intention to propose anything which should in any way interfere with or cover the ground of the committee reporting on methods of analysis.

But, while the association had what might be called a commercial object, he asked if the time was not ripe for the enlargement of its object and its purpose.

He asked the Association to consider how much of the wonderful advance of organic chemistry during the past twenty-five years had been due to the commercial importance of the coloring materials invented by the chemists; but the feeding of domestic animals in the United States and in Europe was of immensely more importance, and the food of man was of still greater moment. He believed that a period was coming in the advance of chemical knowledge in which the most thorough, the most accurate, and the most profound study—biological study—must inevitably be devoted to the matters of food and nutrition. The analytical chemist must call to his aid the organic chemist and the biologist. Research in physical chemistry was needed. The experiment stations found themselves confronted with serious problems, and the question was whether chemistry could do something more to help them out. He was enough of an enthusiast to believe that the work of the experiment stations and cognate work in this line was destined in the not distant future to have a very important influence upon chemical science—an influence at least comparable with that which the color industry had exerted upon organic chemistry. It was with this thought in mind that he had considered the special subject here discussed. What was wanted was a knowledge of the principles underlying the analytical methods and the estimates of the feeding values. There were men who would come to see the importance of this. Some of the best chemists the world over now saw a field for work here. He would like, if the Association thought it perfectly proper, to have a committee appointed which should take the matter into consideration.

Mr. Kedzie said that there were a good many things to be done, and the question was not in regard to their worth and value but in regard to the fitness of the Association for undertaking the work. As an association of agricultural chemists its sphere was very limited and within it distinguished success had been met. The question now was whether the Association should branch out into other fields and enter upon other lines of investigation in addition to those upon which it was now engaged or should leave those to be done by other chemists, or by its own members in other organizations. It seemed to him that the proposed broadening of the field would tend to scatter and weaken instead of concentrating the energy of the Association. He raised the question whether it was within the sphere of the organization to adopt the line of investigation which had been suggested.

Mr. Jenkins said that possibly Mr. Kedzie misapprehended the drift of the motion which did not commit the Association to any scheme for carrying on or directing any work or investigation. The motion was for the appointment of a committee to consider the report upon the desirability of such work, or methods of carrying on such work, without in any way committing the Association.

At the request of Mr. Jenkins the president read the motion.

Mr. Harrington said that it seemed to him that the motion, if carried, would at once broaden the work of the Association. The constitution said:

> The objects shall be: (1) To secure uniformity and accuracy in the methods, results, and modes of estimates of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

He saw no particular objection to the appointment of the committee, but could see no object that such a committee could reach without enlarging the work of the Association, and, unless it did that, it seemed to him that the work of the committee would be useless.

Mr. Nicholson said that he thought Mr. Atwater had really struck the key-note to the future success of the work of the experiment stations and of the analytical chemists in simply calling attention to the absolute necessity of sooner or later getting underneath what now lay upon the surface. If, instead of pulling over the dust-heaps and working upon the piles already accumulated, the Association could get beneath them and engage itself with something that would (if he might use the expression) give a little more life to the work in hand, he thought it would be well. By looking at the experiment stations abroad a lesson might be learned from what they began to do in their first inception and were to do now. Some of their men were doing the very best work in pure chemistry, and that not as a leading part of their labors, but incidentally. While not altogether familiar with the objects of this Association, this being the first meeting he had attended, he looked upon the motion as designed to call the attention of the individual worker to clues which he might discover, and which he should be encouraged to follow up, with a view of discovering what was underlying the work he was doing. He believed this was the most important matter which the Association had to consider.

Mr. Strahan said that he would call attention to the fact that most of the members were connected with scientific institutions of learning and had directing power over a number of gentlemen working in that line. Why could they not without any sacrifice of agricultural work indicate to others under their charge scientific investigations in pure chemistry?

Mr. Harrington said that in a broad way the work of the chemist and of the station employés was laid down in the Hatch bill. He should certainly be opposed to the appointment of a committee with power to prescribe work for any particular chemist at any station. He presumed that each man at the head of an experiment station undertook to get below the surface work, if such had been done, and to investigate these subjects for himself. The line on which these investigations should take place was broadly stated in the Hatch bill, and its specific character, it seemed to him, should be left to the decision of each individual chemist.

Mr. Atwater said that perhaps he ought to make a specific answer to the statement of Mr. Harrington, inasmuch as he was the one man to whom, more than any other, such a statement should be addressed. He thought that his own ideas would be found to accord very thoroughly with those of Mr. Harrington, and did not believe that the motion in question could be made to mean anything contrary to that gentleman's views; if it could he would be glad to have it changed, and would ask Mr. Jenkins to so change it that it should not conflict with the ideas of Mr. Harrington.

Mr. Burney said that he had been particularly isolated for nine or ten years in South Carolina, where it had been necessary to build up the educational institutions from the wreck remaining after the civil war. One like himself found stimulation in contact with the members of the Association, and would enjoy the benefit of just such discussions as would arise from the introduction of the material which would doubtless be presented by this committee. Why not profit by the example of the European experiment stations, which did not hesitate to pursue any line of investigation involving work in pure chemistry? They did not stop at mere methods of chemical analysis. It would be deplorable, it seemed to him, if they did.

The president said that he feared the discussion of the question was trespassing beyond the domain of the Association, which was not an experiment-station organization, but an entirely different thing. It was true that a good many of the members belonged to experiment stations, but the Association should keep constantly before it the fact that its primary business was analytical work. As presiding officer he deemed it his duty to call to the attention of the Association the fact that its functions were laid down and defined in the first article of the constitution. The work proposed to be done was a grand work, and the occasional friction between the station men was good, but the question now before the meeting was very largely a question as to whether the scope of the Association's work should be enlarged. He should be glad to do all that he could to help the thing forward if it was pertaining to the work of the Association. He was not clear in his own mind as to that. Certainly the work ought to be done; all would admit that. He would again read the motion, which appeared to him to be a very excellent good thing, and in connection with it he would read the first article of the constitution.

The president read the motion and article referred to.

Mr. Atwater said that he should be very sorry indeed to know that he had brought anything before the Association which might seem to ask for a change of its methods in any way. He suggested that the motion might be improved by substituting for the word "plans" the words "ways and means for securing."

Mr. Voorhees said that he did not see how the motion interfered in any way with the work of the Association. It seemed to him directly in line with the intention expressed in the constitution, and "to afford opportunity for the discussion of matters of interest to agricultural chemists;" it had direct reference to the analysis and study of the products, the analysis of which the Association met from time to time to consider, and he thought it highly important that the work already being done should be enlarged, if that were necessary, in order to propose and investigate ways and means of study which would be advantageous in the analysis of feeding stuffs. Coming directly under the quoted section of the constitution, he thought it very desirable that the motion should be passed.

The motion, as amended, was read by the president, as follows:

Moved that a committee be appointed to consider the report upon the ways and means for securing more thorough chemical study of foods and feeding stuffs with the purpose of obtaining such information as will aid in the improvement of methods of analysis and in the more satisfactory estimation of nutritive values.

The motion was carried.

The president suggested that in order to save time it might be well to omit from the reading of reports such portions as related to the literature of the subjects concerned and were not essential to the discussion.

Mr. Jenkins presented the following report on potash:

STATEMENT OF THE REPORTER ON POTASH.

Since our last meeting nothing of special value has been published regarding the determination of potash in fertilizers. Woussen (Ann. Agronom., 13, 431; Fres. Zeitschr., 28, 343) proposes to weigh the platinum contained in the double chloride, rather than the double chloride itself, after reducing it with formic acid. He recommends a solution of platinum chloride twice as concentrated as the one used for the Lindo-Gladding method, and for each determination takes enough to yield .75 to 1.00 gram of platinum.

Kretzchmar (Chem. Zeit., 11, 418; Fres. Zeitschr., 28, 95) proposes to use, particularly for ashes and minerals, a method quite like the alternate recommended by this Association, with the following modifications: A solution containing five times the quantity of potash which is to be used for the determination, after precipitation with barium chloride, ammonium carbonate, and ammonium hydrate, is evaporated to dryness, without previous filtration, and heated to 110° C. The dry mass is taken up in water, a few drops of ammonia are added, and it is filtered and washed. This treatment he claims makes the precipitate fine, less flocculent and easier to wash, while without it the separation of potash is apt to be incomplete. In special cases the filtration can be omitted. The filtrate and washings are then brought to 500 cubic centimeters and in an aliquot portion, after evaporation and ignition to expel ammonium salts, potash is determined. If much magnesia is present he prefers to evaporate with oxalic acid, ignite gently, and filter off the magnesium carbonate before adding platinum solution.

A modification of the alternate method is described in the report of the Maine Agricultural Station for 1886–'87, page 123, as follows:

"After the addition of barium hydrate, in excess, with subsequent heating, the solution of ammonium hydrate and ammonium carbonate are added without previous filtration, and without heating the mixed precipitates are allowed to settle, when they are thrown upon a filter and thoroughly washed. This causes a great saving of time as compared with two filtrations, for not only is one filtration saved, but the mixed precipitates filter much more rapidly and wash more easily than the precipitate from barium hydrate. Numerous comparisons of this method with the one involving two filtrations have been made during the past two or three years, with no appreciable difference in results." This modification is also indorsed in the Maine Station Report for 1888.

Your reporter found it quite impossible to prepare and distribute samples for potash determinations till early in February, and perhaps the rather small number of reports received is due to this failure on his part. A circular was sent to all agricultural experiment stations, to some State chemists, and others asking, if each wished samples sent. Eight stations for various reasons declined to participate, and eighteen stations and three State chemists requested samples. To all of these samples were sent, together with a circular describing the nature of the samples. In June reports of the work were asked for by a circular sent to each who took part in it. Twelve sent reports, five found themselves unable to do the work, and four made no reply. The persons who did the analytical work and the letters used to designate each laboratory in the following statement are:

		Analyst.
A	Colorado Agricultural Experiment Station	David O'Brine.
B	Maryland Agricultural Experiment Station	H. J. Patterson.
C	Mississippi Agricultural College	L. G. Patterson. B. W. Kilgore.
D	Vermont Agricultural Experiment Station	J. L. Hills. B. O. White.
E	New Jersey Agricultural Experiment Station	E. B. Voorhees. H. R. Baldwin, jr.
F	Cornell Experiment Station	W. P. Cutter.
G	U. S. Department of Agriculture	A. E. Knorr.
H	University of Georgia	H. C. White.
I	Virginia Department of Agriculture	R. H. Gaines.
J	Texas Agricultural Experiment Station	Duncan Adriance.
K	Pennsylvania Agricultural Experiment Station	W. T. Sweetzer.
L	Connecticut Agricultural Experiment Station	A. L. Winton, jr. T. B. Osborne. R. S. Curtiss.

The samples which were sent and the reports which were received, were as follows:

Sample No. 3 was "chemically pure"—potassium chloride finely pulverized and dried. It contained no appreciable quantity of either insoluble matter, lime, magnesia, or sulphuric acid. It lost .10 per cent. when heated to 130° C. If this was moisture the sample contained 63.08 per cent. of potash, K_2O.

Determinations made by the three chemists in our station laboratory, with specially purified platinum chloride, gave 63.08, 63.09, 62.89, or an average of 63.02 per cent. of K_2O.

Determinations of water made in five different laboratories showed from a trace to .40 per cent. of moisture, an average of .19 per cent. If this was the true percentage of moisture and the salt was otherwise pure, it contained 63 per cent. of K_2O. The determinations made in twelve different laboratories are as follows;

Laboratory.	Determinations.	Remarks.
	Per cent.	
A	63.10	
B	63.16	Average of three determinations.
C	61.92	
	62.08	
D	62.68	
	62.96	
E	63.00	
	63.04	
F	62.91	
	62.94	
G	62.89	Lindo-Gladding method.
	62.98	Do.
	62.81	Alternate method.
	63.07	Do.
H	63.24	
I	63.12	
J	62.92	
K	63.56	Lindo-Gladding method.
	63.54	Do.
	63.18	Alternate method.
	63.18	Do.
L	63.08	
	62.89	
	63.09	

Or 24 determinations all told, excluding K.

K discovered an impurity in his reagents after the results were reported, which threw doubt on the correctness of the determinations. Excluding the results of C, which are unquestionably too low, and of K, the average of nineteen separate determinations is 62.96 per cent. of K_2O, or four-hundredths per cent. lower than is actually present. Eleven of these were within one-tenth per cent. of actual percentage, seven others differed less than two-tenths per cent., while the remaining six may be called "wide."

In six laboratories the results were within one-tenth, in three others within two-tenths of the actual percentage, and in two others the results were "wide." I should regard two tenths a fair and .25 per cent. as a generous margin for unavoidable errors in a determination of this kind.

Nine out of the twelve laboratories came within this limit.

Sample No. 2 was damaged cotton-seed meal. The determinations made in twelve different laboratories were as follows:

Laboratory.	Determinations.	Remarks.
	Per cent.	
B	1.93	Average of three determinations.
C	1.58	
D	1.56	
F	1.22	
G	2.17	Alternate method.
	2.17	Official method.
H	1.64	Average of four determinations.
I	1.67	
J	1.89	
K	1.66	Alternate method.
	1.73	Official method.
L	1.82	Average of three determinations.

Making in all twenty-nine determinations. The average of these is 1.65 per cent.; or if we average only the single average result of each laboratory, 1.71 per cent.

G, by the alternate method which prescribes the destruction of organic matter with oil of vitriol before extracting potash, found 2.17 per cent. of K_2O.

D found 1.89 per cent. by incinerating the substance before extraction, and three determinations made in our own laboratory in the same way gave 1.77, 1.82, and 1.86 per cent., making the average of the four 1.90 per cent.

It has been abundantly proved in past years that it is necessary to char such vegetable products as cotton-seed meal previous to extraction in order to accurately determine either the potash or the phosphoric acid, and I therefore feel justified in taking the higher figure, 1.90 per cent., as correct.

Sample No. 3 was a mixture of weighed portions of samples 1 and 2 with other chemicals. The object was to make a mixture which should offer some special difficulties. One hundred grams of No. 1 pure potassium chloride, 200 grams of No. 2 cotton-seed meal, 125 grams of pure magnesium sulphate made by dissolving washed magnesia in C. P. H_2SO_4 and 75 grams of pure sodium chloride were mixed and sifted together very thoroughly. This mixture contains 13.36 per cent. of K_2O. The circular sent with the samples stated that it contained both cotton-seed meal and potassium chloride. The determinations reported were as follows:

Laboratory.	Determinations.	Remarks.
	Per cent.	
A	14.65	
B	13.39	Average of three determinations.
C	13.66	
D	13.45	
E	13.09	
F	13.71	
G	13.54	Official method.
	13.38	Alternate method.
H	13.14	Average of five determinations.
I	13.52	
J	13.83	
K	13.06	Alternate method.
	13.25	Official method.
L	13.48	

The general average of the average results reported by these twelve laboratories is 13.52, or excluding A, which is furthest from the mean, 13.42. This is .06 per cent. higher than the actual composition of the mixture. Of the fourteen results from twelve laboratories, seven, that is one-half, were within .2 per cent. of the actual percentage present, two others were within .3 per cent., and the others showed greater differences. One was 1.3 per cent. too high.

It will be noticed that in this mixture potash can not be most accurately determined by the official method unless there are compensating errors. The alternate method prescribes ignition with oil of vitriol in case of vegetable substaces like cotton-seed meal, but the Lindo-Gladding does not. The determinations made in the sample of cotton-seed meal indicate that the error arising from incomplete extraction of K_2O might be from .3 to .6 per cent., which in the mixture we are considering would amount to about .12 to .15 per cent.; an error not large, to be sure, but too large to be neglected by any one whose conscience has not been seared by doing slipshod work.

Moreover, this source of error can not be wholly avoided by simple ignition with oil of vitriol. The mixture contains so large a quantity of alkaline salts that it readily fuses and protects the carbon from oxidation while at the same time there is great danger of mechanical loss during ignition by the bubbling of the liquid mass.

The best way to treat the sample is to wash on a small filter with water to remove soluble salts, then ignite the residue with oil of vitriol, dissolve in water, and add to the washings. Such samples as this, it will be urged, are rarely met with. This is true; but it is also true that they are met with, and must be treated as the special cases require. It is noteworthy that while the official method would be expected to give results .15 per cent. lower than the actual percentage, the average of the reported result was a little higher. This, I believe, was caused by incomplete washing of the double chlorides of potasium and platinum with the solution of ammonium chloride.

I recommend the continuance of the present methods of determining potash for the coming year, and suggest that the first sentence under the heading "alternate method," page 60 of last report, be transferred to the previous page under "method of determining potash."

This will indicate that the precaution of charring organic substances like cottonseed meal is to be observed with the Lindo-Gladding method as well as with the alternate.

It is to be regretted that all the agricultural stations can not take part in the analyses of samples supplied to members of this association. That there is abundant need of elementary instruction in the very simple method of separating potash and soda will be apparent to any one who examines the ash analyses of vegetable products made in certain stations.

It is a mistake to suppose that accuracy in fertilizer analyses is more important than elsewhere. I regard it as less important. Errors in fertilizer analysis are more readily detected and corrected than others, and by the end of another season they are forgotten. But the result of a stupid analytical blunder in the analysis of a feeding stuff, or of the ashes of a vegetable product, gets into print, mars tabulated statements, vitiates averages, goes into text-books, and wallows around generally in chemical literature bottom up like a derelict vessel at sea, always an eye-sore, always a danger, and it never sinks.

It was moved that the report be accepted. The motion was seconded.

Mr. Voorhees said that it had been the custom in his laboratory (and he thought that in following that custom the law upon the subject had been properly interpreted) to make all potash solutions in water without previous ignition. The New Jersey law required that solutions of potash should be made in distilled water. He wished to inquire as to the experience of others in regard to the time necessary to extract with water all the potash in the organic substance sent out. He had been informed that more potash was present than he could secure by the method allowed. It seemed to him that if the recommendations of the reporter were included under the Lindo-Gladding method the law in New Jersey would have to be changed or he could not follow the association method, for he did not believe that as the law stood he was permitted to ignite his potash samples before making the solutions. The question of extraction had been brought to their notice by fertilizer firms who claimed that potash was all soluble in water, but it required twelve hours of boiling. He thought that some uniform length of time should be fixed upon for boiling the solution at all the stations. He boiled uniformly for half an hour, but knew that he could not get all the potash out of certain organic substances in that time, and always ignited first when determining potash in fodder plants.

Mr. Frear said that in Pennsylvania also the law provided only for the determination and valuation of potash soluble in water, and made no allowance whatever for potash present in organic combination, as in cotton-seed and other substances which might be introduced in fertilizers. He was sorry that such was the case, but did not feel inclined to force upon the other members his own disability; that would be an injustice to the manufacturers and sellers of fertilizers in other States.

He did not see how there could be any difficulty if it were remembered that analysts working in different States work under different conditions.

Mr. Jenkins said that he was not present at the meeting of the Association last year, and therefore had not realized the difficulty occasioned by the State laws referred to. In the ordinary run of commercial fertilizers the method proposed, simply boiling with water, seemed to him sufficient, and he thought that it ought to be followed. With cotton-seed meal, however, where he had had it pure and simple he had always followed the alternate method, knowing perfectly well that he could not get out the potash by boiling with water. In view of the condition of things reported in several States he felt hesitation in making the recommendation, and it might be better to let the method stand as it was; then those not restricted might, if they chose, use the alternate method where the other was not prescribed. One other point deserved consideration. Ten minutes' boiling was supposed to be sufficient for a superphosphate; he had generally boiled nearer half an hour, though ten minutes was sufficient in nearly all cases. He was therefore in favor either of making a change under superphosphates or, leaving that as it was, in inserting in the directions for sulphate of potash and kainite "dissolve by boiling with water for half an hour."

Mr. Voorhees said that a question as to the agricultural value of the form of potash seemed to be involved. He would ask whether in affixing a commercial value the potash extracted by simply boiling in water should not have a higher price attached than that extracted after ignition? Whether the effect of the same percentage of potash soluble in water, say by one-half hour boiling, from organic compounds and from potash salts would not be the same was a point that should be considered.

It was moved to adopt the recommendation of the reporter on potash as amended, that under the head of sulphate of potash, kainite, etc., the words "boil for ten minutes" be striken out and that the words "boil for half an hour" be inserted in their place.

The motion was seconded and carried.

Mr. McDonnell said that a method shorter even than the alternate method, and sufficiently accurate for any purpose, was to merely boil the fertilizer with water, making it up to the mark, then shaking and allowing it to cool and settle for a few minutes; then add a heated solution of barium hydrate, and, after solution, add ammonium carbonate; add all together, filter and add hydrochloric acid. In most cases this gave a very fair result. He had made some analyses in that way, but had no figures to offer. However, it compared very favorably with the longer method in nearly all cases.

Mr. Gascoyne said that Mr. Wiley had published in a bulletin some years ago certain experiments on that point. By using one filtration for the double precipitation the results were lower than by using the regular method, that is the alternate method now.

Mr. Frear said that his attention had been called to the fact that the method spoken of was in use at the Maine station, and the claim was made that the filtration was more rapidly conducted by that method, while the results obtained were practically the same whether two filtrations were made or only one. He thought it would be of interest to study this matter a little further.

Mr. Gascoyne said that Mr. Wiley's results were rather low. It was almost impossible to wash the potash entirely out of the precipitate.

Mr. Strahan said that prior to the adoption of the Lindo-Gladding method the process mentioned was always used in his laboratory. He, like Mr. Wiley, had found the results low as compared with those given by the other method. He had found, moreover, that it was always necessary to evaporate in a platinum dish and ignite twice, in order to remove all the organic matters and ammonium salts.

It was moved that the method of determining potash recommended by the reporter, and as subsequently modified, be adopted.

The motion was seconded and carried.

Mr. Scovell presented the following report on the determination of nitrogen.

REPORT OF M. A. SCOVELL, REPORTER ON NITROGEN.

As your reporter on nitrogen, I have the honor to herewith present my report.

The report covers: (1) Brief notices of analytical methods for determining nitrogen proposed since our last convention. (2) Results of work done by members of Association and others. (3) Recommendations.

NOTICES OF ANALYTICAL METHODS PROPOSED DURING THE YEAR ON THE DETERMINATION OF NITROGEN.

[Compiled by A. M. Peter, Kentucky Agricultural Experiment Station.]

Kjeldahl and modifications.

L. L'Hôte (Comptes Rendus, 108, 59–62 and 817–20) states that the Kjeldahl method gives lower results than the soda-lime and absolute methods, and thinks that some ammonium sulphate is volatilized during the reaction, and that the decomposition of the organic matters is incomplete. In his opinion the method does not afford the simplicity, accuracy, and dispatch of the soda-lime method. The author's experiments show that with asparagin, caffein, urea, uric acid, and oxamid the decomposition with sulphuric acid is easy and complete, and all the methods give correct and concordant results. With brucine and cinchonine and many agricultural products, however, the decomposition is not complete, even after a long time, and with these the Kjeldahl method gives too low results.

P. Cazeneuve and L. Hugonenq (Bull. Soc. Chim., 49–991) state that the nitrogen of urine can not be correctly determined either by Kjeldahl or soda-lime methods, but only by the absolute.

C. Violette (Comptes Rendus, 108, 181–83) obtains concordant results by all three methods, and attributes the low results obtained by L'Hôte to the use of mercury as well as to the causes assigned by him. On this account the author uses Nordhausen sulphuric acid, and digests eighteen to twenty-four hours without mercury.

J. W. Gunning (Zeitschr. An. Chem., 28, 188–91; also abstract in Analyst, vol. 14, p. 79). The author uses a mixture of one part potassium sulphate and two parts strong sulphuric acid; 0.5–1.0 gram of substance is heated in a 300 cubic centimeter round-bottom, short-neck flask with 20 to 30 cubic centimeters of the above mixture until frothing ceases. A funnel is now put into the mouth of the flask and covered with a watch-glass, and the heating continued till the contents of the flask are colorless, which requires one-half to two hours, generally about one hour. The distillation is conducted as usual, but the author prefers to standardize his acid by the iodine and hyposulphite method. Test analyses gave for uric acid 33.3 per cent. N.; theory, 33.33; aniline oxalate, 10.0 per cent.; theory, 10.1 per cent. N.

Otto Foerster (Chem. Zeit., 12, 1555 and 13, 229) uses sodium hyposulphite in the place of zinc dust. The solution of the nitrate, corresponding to 0.5 gram KNO_3, is evaporated in the digestion flask, and when dry, treated with 15 cubic centimeters of 6 per cent. phenolsulphuric acid, and after it is dissolved 1 to 2 grams sodium hyposulphite are added. The flask must be cooled during this operation or the results will be a little low. After the decomposition of the hyposulphite 10 cubic centimeters strong sulphuric acid and a little mercury are added, and the flask heated until oxidation is complete, which requires about a half hour. The distillation is as usual except that the author uses a bulb-tube arrangement as receiver.

In fourteen experiments with pure potassium nitrate the results ranged from 13.79 to 13.91 per cent. nitrogen.

A. Stutzer (Landw Vers. St., 35, 445–48). The author's method of carrying out the Jodlbaur method is as follows: 0. 5 gram nitrate or 1 gram fertilizer containing nitrates is rubbed up with 2 to 3 grams ignited gypsum, in order to divide and dilute the nitrate. The mixture is brought into a flask of about 350 cubic centimeters capacity, and 25 cubic centimeters of 4 per cent. phenolsulphuric acid are added and well mixed by shaking. After five minutes the flask is cooled and 2 to 3 grams zinc-dust are added gradually and two drops of mercury. The digestion is finished as usual and the distillation is performed in the same flask.

Georg. Roch (Pharmazeut. Centralhalle, 30, 48–9) performs the distillation in the Kjeldahl method without a condenser, using a retort with inclined neck and receiving the distillate in two Erlenmeyer flasks containing standard acid.

Otto Lange (Chem. Zeit., 12, 1587) combines the determination of total phosphoric acid with that of nitrogen, as follows: 10 grams of substance with ½ to 1 gram copper sulphate are decomposed with sulphuric acid as in the Kjeldahl method. When the reaction is finished, which requires two to three hours, the solution is diluted, cooled, and made up to 500 cubic centimeters. The ammonia is determined in 50 cubic centimeters of the solution by distillation with NaOH as usual, and the phosphoric acid in another 50 cubic centimeters by precipitation with magnesia mixture in presence of citrate of ammonia. When sulphate of copper is used instead of mercuric oxide it is unnecessary to use potassium sulphide in the distillation.

T. M. Drown and H. Martin (Jour. An. Chem. 3, 258; also Chem. News, 59, page 272), and Henry Leffman and William Beam (Am. Chem. Jour., and Chem. News, 59, page 310). The authors apply the Kjeldahl method to the determination of organic nitrogen in potable waters. Results satisfactory.

M. Zecchini and A. Vigna (Staz. Sper. Agr. Ital., 15, 298–304). The authors call attention to the danger of loss of ammonia on adding NaOH to the solution in an open flask, and recommend the use of a funnel tube. They use a little powdered zinc in the distilling-flask and pass a current of air through the flask during the distillation to prevent bumping.

G. Lunge (Zeit. f. Angw. Chem., 88, 661–2) shows that the method of purifying sulphuric acid by boiling with a little potassium nitrate is entirely useless. The author showed by experiment that the excess is not expelled even after long boiling.

W. M. Stein and P. W. Schwartz (Jour. An. Chem., 3, 136) describe a new apparatus for determination of ammonia by distillation, as in the Kjeldahl method. To the distilling-flask is attached a pear-shaped bulb into which the tube from the condenser passes. The end of this tube is drawn out slightly but not closed, and turned to one side within the bulb, and is provided with three side openings for the passage of the steam to the condenser. The receiver is an Erlenmeyer flask with a side tube filled with fragments of broken glass through which the standard acid is introduced. This apparatus is said to prevent entirely the passage of spray into the condenser.

Wrampelmeyer (Chem. Zeit., 12, 1317) describes an apparatus for automatically measuring out .05 cubic centimeters of mercury for the Kjeldahl determination.

J. Kjeldahl (Abstract and Figure in Chem. Centlb., 59, 1373) describes a bulb tube for the distillation in which the vapors are passed through boiling water before reaching the condenser.

E. Aubin and L. Alla (Comptes Rendus, 108, 246–48 and 960–61) in opposition to L'Hôte, the author prefers the Kjeldahl method to the soda-lime, and maintains its accuracy. They use .5 gram substance, .5 gram mercuric oxide, and 20 cubic centimeters sulphuric acid.

M. J. Bartlett (annual report of the Maine State College Agricultural Experiment Station, for 1888, page 205): At the Maine station a modification of the Jodlbaur method is used when nitrates are present and the results have been entirely satisfactory. The phenolsulphuric acid is allowed to stand on the substance ten minutes at the ordinary temperature before adding the mercury. "The only precautions necessary to secure good results are : To mix the substance with the acid and phenol thoroughly before adding zinc or heating, and to have the flame very low on beginning to heat. Twenty-nine test analyses by this method are given, the results of which are very satisfactory. Four analyses of potassium nitrate give 13.75 to 13.93 per cent. nitrogen; mean, 13.84 ; theoretical, 13.86.

At the New Jersey Agricultural Experiment Station (annual report for 1888, page 131–2) a comparison has been made between the Kjeldahl and the soda-lime methods, and also between the salicylic acid modification of the Kjeldahl and the absolute methods. No figures are given for the Kjeldahl and the soda-lime methods, but we find the following statement: "Of one hundred and thirty-four samples of complete fertilizers, eighteen samples of high-grade nitrogenous material, and nineteen samples of ground bone, the average results in total nitrogen are practically identical by both metods." The results are given of the analyses by the Scovell and absolute methods of forty-two fertilizers containing nitrates. In these the total nitrogen varied from 0.67 to 7.40 per cent.

In sixteen instances the Scovell method gave from .01 to .12 per cent. more nitrogen than the absolute method, the average excess being .06 per cent. In twenty-five cases the results were from .01 to .15 per cent. below those obtained by the absolute method; the average deficit being .07 per cent. In twenty-one analyses, or 50 per cent. of the whole, the difference is less than .06 per cent., "the average differences by the two methods being less than .02 per cent." All results were corrected by blank experiments with the re-agents.

Determination of nitrates.

H. Wilfarth (Zeitschr. Anal. Chem., 27, 411–33). Details are given of the author's modification of the Schloesing method for the determination of nitric acid, in which the oxidation to N_2O_5 is accomplished by means of hydrogen peroxide. Apparatus and method are fully described in the original. The results are higher than by the original method, except in presence of organic substances.

A. Devarda (Zeitschr. f Zuckerindustrie, 17, 609) recommends as the simplest method of determining nitrogen in Chili saltpeter to determine the chlorine in the original substance, and again after conversion into chlorine by repeated evaporation with strong hydrochloric acid and drying at a high temperature to remove all free hydrochloric acid. From the difference the amount of nitrogen is calculated. Results are accurate. To apply the same method to the determination of nitrates in superphosphates the author boils the water solution of superphosphate with excess of pure zinc oxide and uses a portion of the filtrate for the evaporation with HCl.

Bailhach (Comptes Rendus, 108, 1122, also Chem. News, 59, page 293). Author describes a method of determining nitrates by their action on ferrous sulphate solution and titration. Results given are for pure sodium nitrate and Chili saltpeter and show good agreement with theory and with the Schloesing-Grandeau method. The solutions used are standard ferrous sulphate solution containing 100 grams $FeSO_4$ and 75 cubic centimeters, conc. sulphuric acid per liter. Bichromate of potash standard solution containing 17.85 grams bichromate per liter, and standard solution C. P. sodium nitrate, containing 50 grams of that salt per liter, and a solution of sodium hydrocarbonate saturated in the cold. The apparatus is a flask graduated to 250 cubic centimeters with rubber stopper, through which passes a funnel-tube reaching to near the bottom of the flask and a short exit tube drawn out to a narrow opening.

The cylindrical funnel-tube should hold 35 to 40 cubic centimeters, and is provided with a stop-cock. Fifty cubic centimeters of the ferrous sulphate solution are put into the flask with 25 cubic centimeters of conc. sulphuric acid, the stopper inserted, and the flask placed on a sand-bath. Twenty-five cubic centimeters of the sodium hydrocarbonate solution are gradually run into the flask by means of the funnel-tube, and the contents of the flask heated to boiling. Before all has run in, 10 cubic centimeters of the sodium nitrate solution are put into the funnel, and the stop-cock so adjusted that the boiling and the evolution of the gas are steady and continuous. After this 25 cubic centimeters sodium hydrocarbonate solution are again run in as at first and the flask allowed to cool. The cool solution is made up to the mark and an aliquot part titrated with the bichromate solution to determine the amount of ferrous sulphate remaining. From the difference the amount of nitrate is calculated.

Absolute method.

P. Cazeneuve and L. Hugounenq (Bull. Soc. Chem., 49, 900–901), recommended the use of manganese carbonate in the tube as a source of CO_2. It is preferable to bicarbonate of soda, as the change of color shows the progress of the decomposition and it does not re-absorb any carbon dioxide on cooling.

Richard Zsigmondy (Zeit. Anal. Chem., vol. 28, pt. 1). In analyzing a nitrogenous organic compound containing chlorine, the author obtained a considerable excess of nitrogen over the theoretical. The author thinks the error was due to the formation of oxychloride of copper which was subsequently decomposed, giving up its oxygen, which was measured as nitrogen. The error may be avoided by passing carbon dioxide over the copper oxide while hot, and letting it cool in a current of the same gas.

Soda-lime method.

Quantin (Bull. Soc. Chim. 50, 198–201) discusses the part taken by the soda-lime in the conversion of the organic nitrogen into ammonia. Such nitrogenous organic substances as are analyzed by the soda-lime method, when heated alone with exclusion of air, would yield the greater part of their nitrogen in the form of ammonia, the rest being left in the carbonaceous residue. The author thinks that the function of the soda-lime is to decompose this carbonaceous residue, and that in this decomposition an alkaline cyanide is formed which is subsequently decomposed by the vapor of water yielding ammonia and a carbonate.

According to this view a low heat at first with intense heating at the close of the combustion should give the best results.

RESULTS OF WORK DONE.

Last May your reporter sent to nineteen official and seven commercial chemists three samples for nitrogen determination, also small bottles of standard hydrochloric acid and zinc dust, and inclosed in a letter a blank for tabulating results of analyses, and comparison of standard acid. The samples were marked 1, 2, and 3, and their composition was as follows:

		Grams.
No. 1.	Sodium nitrate (containing 16.39 per cent. of nitrogen)	607
	Sodium chloride	18.2
	Calculated per cent. of nitrogen	15.91

		Grams.
No. 2.	Cotton-seed meal.	
No. 3.	Sodium nitrate (dried at 100°)	9.00
	Cotton-seed meal	18.50
	Ammonium sulphate (dried at 100°)	3.10
	Acid phosphate (free from nitrogen)	64.40
	Muriate of potash (free from nitrogen	5.00
		100.00
	Calculated per cent. nitrogen	3.55

Results have been received from fifteen laboratories, covering the work of seventeen chemists.

RESULTS OBTAINED.

Kjeldahl method.

Analyst.	No. 2.
	P. ct. N.
F. B. Carpenter (North Carolina Experiment Station)	7.58
J. L. Hill (Vermont Experiment Station)	7.61
B. O. White (Vermont Experiment Station)	7.54
A. A. Bennett (Iowa Agricultural College)	7.31
C. B. Collingwood (Arkansas Experiment Station)	7.63
William Frear (Pennsylvania Experiment Station)	7.31
H. S. Grindley (Experiment Station, University of Illinois)	7.50
R. C. Kedzie (Michigan Agricultural College Experiment Station)	7.47
J. T. Willard (Kansas Experiment Station)	7.49
J. M. Bartlett (Maine Experiment Station)	7.61
D. H. Merrill (Maine Experiment Station)	7.65
Charles Gibson (Northwestern Fertilizing Company)	7.44
W. M. Saunders (Brown University)	7.49
T. C. Trescot (U. S. Department of Agriculture)	7.25
M. A. Scovell (Kentucky Agricultural Experiment Station)	7.58
Average	7.50

Kjeldahl modified for nitrates.

Analyst.	No. 1.	No. 3.
	P. ct. N	*P. ct. N*
F. B. Carpenter (North Carolina Experiment Station)	15.89	3.46
J. L. Hill (Vermont Experiment Station)	15.90	3.63
B. O White (Vermont Experiment Station)	15.87	3.53
A. A. Bennett (Iowa Agricultural College)	16.41	3.64
C. B. Collingwood (Arkansas Experiment Station)	15.45	3.64
Wm. Frear (Pennsylvania Experiment Station)	15 89	3.73
H. S. Grindley (Experiment Station, University, Illinois)	15.74	3.43
R. C. Kedzie (Michigan Agricultural College Experiment Station.)	15.74	3.47
J. T. Willard (Kansas Experiment Station)	15.80	3 51
J. N. Bartlett (Maine Experiment Station)	16.16	3.60
D. H. Merrill (Maine Experiment Station)	16.09	3.60
Chas. Gibson (North Western Fertilizing Company)	16.44	3.23
W. M. Saunders (Brown University)	......	3.40
T. C. Trescot (U. S. Department Agriculture)	15.68	3.65
M. A. Scovell (Kentucky Agricultural Experiment Station)	15.83	3.54
Average	15.92	3.54

Ruffle method.

Analyst.	No. 1.	No. 2.	No. 3.
	P. ct. N	*P. ct. N*	*P. ct. N*
F. B. Carpenter (North Carolina Experiment Station)...	15.83	7.56	3.50
William Frear (Pennsylvania Experiment Station).....	15.93	....	3.72
R. H. Gaines (Virginia Department Agriculture)........	16.16	....	3.67
W. M. Saunders (Brown University)....................	....	7.28	3.27
T. C. Trescot (U. S. Department Agriculture)............	15.62	....	3,44
Average......	15.88	7.42	3.52

Soda-lime method.

Analyst.	No. 2.
	P. ct. N
Wm. Frear (Pennsylvania Experiment Station)..........	7 32
W. L. Snyder (Michigan Carbon Works)..................	7.60
W. M. Saunders (Brown University)	7.43
T. C. Trescot (U. S. Department Agriculture)............	7.06
R. H. Gaines (Virginia Department Agriculture)	7.42
Average	7.37

Table of averages.

Method.	No. sample.	No. of analysts.	Average.	Highest.	Lowest.	Difference.
Kjeldahl	2	15	7.50	7.65	7.25	0.40
Kjeldahl modified for nitrates...	1	14	15.92	16.44	15.45	0.99
	3	15	3.54	3.73	3.23	0.5
Ruffle...........................	1	4	15.88	15.93	13.16	2.77
	2	2	7.42	7.56	7.28	0.28
	3	5	3.52	3.72	3.44	0.28
Soda-lime........................	2	4	7.35	7.60	7.06	0.54

The following table is given to show the percentage of results differing from the average from 0.1 to 1.00 per cent.:

Method.	No. sample.	Percentage of results differing from average not more than—				
		0.10	0.25	0.50	0.75	1.00
Kjeldahl........................	2	60	100	......	......	......
Kjeldahl modified for nitrates..	1	36	80	100	......	......
	3	67	95	100	......	......
Ruffle..........................	1	0	0	25	50	75
	2	0	100	......	......	......
	3	40	100	......	......	......
Soda-lime.......................	2	50	75	100	......	......

REMARKS.

The soda-lime method seems to have been laid aside by active workers, judging from the reports sent in, as only four chemists reported results by this method, and three of these reported results by other methods as well. Of course this method was only used on sample No. 2 cotton-seed meal. The average of the results obtained is

0.15 per cent. below the average by the Kjeldahl method. On samples rich in nitrogen, such as cotton-seed meal, there seems to be great liability of obtaining results too low, unless small quantities of the substance are used and due care exercised in the combustion.

Five chemists report results by the Ruffle method. On sample No. 1 the average is 15.88, the theory 15.91 ; a difference of 0.03 per cent.

For sample No. 2 (cotton seed meal) the Kjeldahl method was used by fifteen chemists, the average of the results giving 7.50 per cent. nitrogen. The results are highly satisfactory ; nine coming within 0.1 per cent. of the average, and all within 0.25 per cent.

Fifteen of the seventeen chemists used the Kjeldahl method modified for nitrates for samples Nos. 1 and 3. On sample No. 1 the average of all the results is 15.92, the theory 15.91. On sample No. 3 the average is 3.54, the theory 3.55.

The average on sample No. 1 includes three results, which are nearly 0.5 above the theory. In making conclusions as to the accuracy of the methods it would be better to leave these three results out of the average, as they are so far out of the way as to reflect inaccuracy or carelessness on the part of the chemist rather than the method. Doing so, the average per cent. of nitrogen found in sample No. 1 is 15.86; a result which is 0.05 per cent. below the calculated amount. The highest result is 16.16 per cent., the lowest 15.74 per cent., if we do not include the result from Arkansas, which we should not, as zinc filings were used instead of zinc dust in this determination. The difference between highest and lowest results is 0.42 per cent., or a difference of only 2.6 per cent. of the average. Fifty-five per cent. of the analysts come within 0.1 per cent. of the average, and 91 per cent. within 0.25 per cent.

The results obtained prove, I believe, conclusively that the modified Kjeldahl method is an accurate and reliable one for the determination of nitrogen in fertilizers containing nitrates. On pure nitrates the results are invariably a trifle low. The loss seems mostly to occur when the zinc dust is added. For this reason zinc dust is objectionable. It is objectionable also in giving so much zinc sulphate to contend against in the distillation. Dr. Burney states that the great amount of salts present causes his flasks to break frequently in the distillation. This has also caused us trouble to some extent. Working on the theory that that H_2S would complete the reduction as well if not better than nascent hydrogen, Prof. A. M. Peter and I made a series of experiments, using zinc sulphide in place of zinc dust. The results on pure potassium nitrate containing a trace of water, giving the theoretical per cent. of nitrogen 13.83, were as follows:

On pure potassium nitrate.

[1. Series.—Three grams zinc dust used.—Results.]

	Per cent.
Nitrogen found	13.76
	13.71
	13.79
	13.78
	13.77
	13.77
	13.69
	13.79
	13.79
Average	13.76
Theory	13.83
Loss	.07
Difference between highest and lowest results	.01

On pure potassium nitrate—Continued.

[2. Series.—Two grams zinc sulphide used in place of three grams zinc dust.—Results.]

	Per cent.
Nitrogen found	13.78
	13.77
	13.80
	13.79
	13.83
	13.80
	13.85
	13.80
	13.72
	13.85
Average	13.80
Theory	13.83
Loss	.03
Difference between highest and lowest results	.13

On pure sodium nitrate.

[1. Series.—Three grams zinc dust.—Results.]

	Per cent.
Nitrogen found	16.36
	16.41
	16.40
	16.42
Average	16.40
Theory	16.47
Loss	.07

[2. Series.—Two grams zinc sulphide in place of three grams zinc dust.—Results.]

	Per cent.
Nitrogen found	16.41
	16.44
	16.41
	16.41
	16.41
	16.39
Average	16.41
Theory	16.47
Loss	.06

RECOMMENDATIONS.

I would recommend that the same methods used this year be the official methods for the ensuing year, with some slight modifications, as follows:

1. As shown by the factors sent the reporter—the standard acid, while closely agreeing in many cases; varies somewhat in others. In order to correct any inaccuracies, if possible, in testing the acid it is recommended that the paragraph describing the standardizing the acid be so changed as to read as follows:

(1) Standard hydrochloric acid whose absolute strength has been determined

(*a*) By precipitating with silver nitrate and weighing the silver chloride, as described in Crooke's Select Methods, page 571.

(*b*) By potassium tetroxalate, which has been purified by recrystalizing three times.

(*c*) By sodium carbonate.

(*d*) By determining the amount neutralized by the distillate from a weighed quantity of pure ammonium chloride boiled with an excess of sodium hydrate.

Half normal acid, *i. e.*, containing 18.23 grams hydrochloric acid to the litre, is recommended.

2. Under the Kjeldahl method, page 63, Bulletin No. 19, Division of Chemistry, U. S. Department of Agriculture, under "(6) granulated zinc," add "or pumice stone, to be added to the contents of the flask in distillation when found necessary in order to keep from bumping."

3. On page 65, under "distillation," third line, after "granulated zinc," add "or pumice stone if found necessary in order to keep contents of flasks from bumping."

In the Kjeldah method, modified for nitrates, it is recommended to use 2 grams of zinc dust instead of 3 grams as hitherto. A number of determinations have been made with 2 grams of zinc dust, and in every case the results show that the amount is sufficient.

In the case of sample No. 1 several determinations with 3 grams of zinc dust gave an average loss of 0.11 per cent. N., while parallel experiments with 2 grams zinc sulphide gave results as follows:

	Per cent.
Nitrogen found	15.84
	15.77
	15.84
	15.81
Average	15.81
Theory	15.91
Loss	.10

The object of course is to use as little zinc dust as possible to avoid excess of salts. With 1 gram of zinc dust the reduction is not complete, as may be seen by the following results:

One gram zinc dust used; per cent. nitrogen found; sample 1.

	Per cent.
Nitrogen found	15.40
	15.42
Average	15.41
Theory	15.91
Loss	.5

It is recommended, also, that the reporter, when sending out samples for next year's determinations, request chemists to try zinc sulphide in place of zinc dust. In such case, 1 gram salicylic acid and 2 grams zinc sulphide are the proportions necessary.

It was moved that the report be accepted. The motion was carried.

At 12.30 a recess was taken until 2 o'clock.

AFTERNOON SESSION, WEDNESDAY.

The convention was called to order by the president at 2 p. m.

The president stated that the reporter on cattle-food analysis wished to bring up one point which had been omitted in the discussion of yesterday; if there was no objection the floor would be accorded to him for this purpose.

Mr. Caldwell said he would recommend the following:

> That the reporter on the analysis of cattle foods be requested to send out a special sample of a cereal preparation to be analyzed by the Dragendorff method, any directions for the same to be sent by the reporter with the sample.

It was moved that the recommendation of Mr. Caldwell be accepted and adopted.

The motion was seconded and carried.

The president said that in the resolution adopted in the morning session providing for the appointment of a committee to consider the report upon ways and means for more thorough chemical study of foods and feeding stuffs the number of persons composing such committee was not stated. It would probably be well to attend to that point now.

Mr. Jenkins moved that the said committee should consist of five persons, to be appointed by the president.

The motion was seconded and carried.

The chair appointed Messrs. Atwater (chairman), Caldwell, Jenkins, Jordan, and Wiley.

The president stated that the discussion of the report upon nitrogen was now in order. The reporter had made certain recommendations which would be taken up in order and passed upon by the association.

Mr. Scovell said that it was recommended to insert after the words "silver chloride," on page 63, under the head of "Kjeldahl method," second line, the words "as described in Crookes' Select Methods (last edition), page 571."

He would suggest that the determinations be made by precipitating and weighing the silver chloride, and that this be done in the manner described in Crookes' last edition.

It was moved that the recommendations of the reporter be adopted.

The motion was seconded and carried.

Mr. Scovell said that he would also recommend, referring still to page 63, the leaving out of (*b*) and (*c*) down to "sodium hydrate," and the insertion in place thereof of the following: "(*b*) by potassium tretroxalate, which has been purified by recrystallizing two or three times; (*c*) by sodium carbonate, as described in Fresenius' Quantitative Analysis, second American edition, page 680."

Mr. Jenkins said that he would not oppose the introduction of the tretroxalate, and did not know that it was necessary to retain the others, but it seemed to him that there was a certain advantage in it. The standard acid was the basis for all operations, and too much care could not be taken in establishing that standard very accurately and beyond the preadventure of a doubt. A coast-survey man would hardly be willing to depend upon one or two observations for the length of his base line, and no one should be satisfied with a single method or with two methods if more can be employed in so important a matter as this. He always used four or five methods and compared them on his acid, and until the results agreed perfectly he did not feel safe.

Mr. Scovell said that he did not believe that the first of the methods was equally accurate with the other two, and he thought that two accurate methods were certainly better than three methods, one of which was not accurate. In sodium carbonate the results were found to be lower, and on that account it might be well to go on and say use calcium carbonate in place of sodium carbonate. He considered the sodium-carbonate method an inferior way of getting at the required standard.

Mr. Jenkins moved that instead of omitting the words recommended by the reporter to be omitted the same be retained and the words "and tretroxalate of potassium" be inserted.

Mr. Caldwell said that he would prefer to have the tretroxalate put second, before the carbonates, as he believed it to be a very safe and easy method of testing.

Mr. Scovell accepted the amendment.

The motion was seconded and carried.

Mr. Scovell said that he would recommend, referring to page 63, under the head No. 6, the substitution of pumice-stone in the place of granulated zinc. He did not insist that pumice-stone be adopted, but would simply recommend it as being better than zinc. He had used granulated zinc in distillation, and had taken about a dozen blanks with some soda which he had and found that the blanks did not agree perfectly; by leaving out zinc and putting in pumice-stone in the manner described by Mr. Wiley the blanks were so even that he could not tell the difference in the sodium neutralized, except in one case he got one cubic centimeter deci-normal soda more. He feared that it might break more flasks than the zinc would. It seemed to bump around, although he would not be certain in regard to that. He would like to ask whether others had experience in that direction. Personally he thought pumice-stone much better than granulated zinc. However, he would change his recommendation, and would say for this year zinc or pumice-stone.

Mr. Voorhees said that he had found it unnecessary to use zinc in his distillations, and supposed that a great many others would say the same. He found when he had used zinc that no better results were obtained; therefore, he had discarded it entirely. For two years he had used no zinc in the final distillation; his distillations were very good, indeed, and he did not think that he had lost a flask this year from bumping of solutions.

Mr. Caldwell said that he thought it unfortunate to be under the necessity of using zinc, and believed that granulated zinc was not nearly so good for that purpose as zinc foil rolled out very thin, cut in narrow strips, and rolled into short spirals. He had used these spirals in his laboratory; they were very much lighter, afforded more points for the steam, and there was far less danger of breaking the flask. He thought it very much better to exclude zinc altogether.

Mr. Voorhees stated that in the last two years his flasks had been made to order; they were round bottom flasks and were quite heavy; something might be due to that, but previously he had distilled without the heavy flasks and found no trouble then. This year he had adopted the method of digesting and distilling in the same flasks, and therefore got the flasks a trifle heavier. He had no trouble from breakage, either in digesting or distilling.

Mr. Scovell asked how much water was put in.

Mr. Voorhees replied that the flask held 500 cubic centimeters, and that he had put in from 350 to 400 cubic centimeters. He boiled for half an hour, and evaporated off from 125 to 150 cubic centimeters. He supposed the final solution would be somewhere near 125 cubic centimeters. Perhaps, on the average, not quite so much as that. He distilled off into flasks holding 250 cubic centimeters, and they were not as a usual thing quite half full, but he had never measured them. He had not accepted this method until a series of determinations had been made proving that there was no loss in it. He had taken duplicate samples, a series of 40 or 50, duplicating by digesting in the same flask over and over again, in order to see how long it could be used for both purposes; and he had found that his results agreed perfectly, that the flasks were as little affected by the digestion as distillation. In modification of Kjeldahl he had found some trouble; the digestion of some samples seemed to affect the flasks, which showed checks around the bottom after one or two determinations. He found the plan of digesting in different flasks cheaper than to use the same flasks for both purposes on samples containing nitrates.

Mr. Scovell said that he would like to add something to his recommendation; he wished it to read "granulated zinc or pumice-stone, when necessary to prevent bumping in distillation."

It was moved that the recommendation of the reporter as amended be adopted.

The motion was seconded and carried.

Mr. Scovell said that he had no further recommendations to make as to the method of analysis. At the proper time he would make a motion that the reporter be requested to ask at least some of the analysts to whom he sent samples to try the zinc suplhide in place of granulated zinc, and see how it came out in their hands.

Mr. Voorhees said that to a certain extent this year he had used metallic mercury instead of the oxide, and had found it very much easier to handle, while its action seemed to be better, the oxidation being more rapid. He had a little arrangement for weighing off .7 of a gram, giving that quantity for each determination. He supposed there would be no objection to that deviation from the method as stated.

Mr. Scovell said that the method for that was described by some authority who had a little hole bored in the stop-cock, every turn of which gave .05 of a cubic centimeter.

Mr. Voorhees said he added caustic potash until the precipitate settled, leaving a clear solution.

Mr. Burney said that if the whole subject were before the meeting he would like to mention the fact that in searching for the cause of the breakage of distilling flasks it had occurred to him that the large precipitate formed by the zinc might be the source of trouble. He had therefore looked for some substitute for the zinc dust. In using the modified Kjeldahl method it had seemed to him that inasmuch as sodium hypophosphite or any hypophosphite when heated yielded PH_3, phosphoretted hydrogen, hypophosphites might well take the place of the zinc dust. He used pure sodium hypophosphite, the best he could get, and used 2 grams at a time; he found that it worked admirably. The difference in favor of the sodium hypophosphite was that the results were just a little higher, a few hundredths of a per cent. He had tried the method on pure nitrates and on a mixture of nitrate with chlorides, thinking that that was one of the most difficult of mixtures to analyze, and the results in his hands were as satisfactory as results could be. There were two or three advantages; there was of course no precipitate formed by the sodium hypophosphite; the solution remained perfectly clear. In addition to that, the digestion was slightly shorter. These were clear gains. Sodium hypophosphite could be obtained pure and need not contain any ammonia, while zinc dust sometimes did. Morever, hypophosphite acts slowly and consequently reaches and reduces all of the nitro-compounds present, which is not always the case with the zinc. It seemed to him that the sodium hypophospite differed radically on theoretical grounds from zinc sulphide or sodium hyposulphite in its action. In view of his experience with this substance he thought that it should be included with the zinc sulphide in the tests made on the official samples to be sent out, and he thought it would be found that the pure sodium hypophosphite answered the purpose precisely and would give uniform satisfaction wherever it was tried.

Mr. Jenkins said that he had been much interested in Mr. Burney's remarks and trusted that the results could be put into shape for publication in the report this year so that the Association could have the benefit of them. He fully agreed that the trial suggested should be made upon the samples sent out. He thought that all chemists recognized the trouble with zinc.

Mr. Scovell moved that in the process of digestion for this year 2 grams of zinc dust be used instead of 3 grams.

The motion was seconded and carried.

Mr. Scovell said that as many had asked where the proper soda for the hydrate could be obtained he would request Mr. Jenkins to inform the members.

Mr. Jenkins said that the kind he used was 98 per cent. granulated caustic soda, sold by the Troy Laundry Machinery Company, 32 Dey street, New York City. This article came in 10-pound drums and cost 8 cents a pound by the single drum, or 7.5 cents a pound if bought in considerable quantities, and was in all respects perfectly satisfactory. It was easily handled, being granular. He also bought caustic potash put up by the same company, equally good in quality.

It was moved and seconded that the scheme of analysis recommended by the reporter on nitrogen, as modified by the amendments proposed and made, be adopted as a whole.

The motion was seconded and carried.

Mr. Burney said that he desired to refer for a moment to the subject of phosphoric acid. He had not attended the meetings hitherto, but it seemed to him that the experience of all present was similar to his own in regard to oxidation. He dissolved the fertilizers in nitric acid, with a little hydrochloric acid, and had repeatedly tested the length of time necessary for the clarification of the solution by the destruction of the organic matter present. He had boiled some solutions for many hours without their becoming clear. Some one had very correctly said that nitric and hydrochloric acids acting upon filtering paper would not destroy it for a considerable time; that was certainly true according to his own experience. He described an experimental method he had used for the oxidation, and stated that the method with nitric and hydrochloric acid had not always proved satisfactory in his hands, as it would not promptly destroy organic matter.

The substance to be oxidized and dissolved was brought into a Kjeldahl digestion flask, 10 cubic centimetres concentrated sulphuric acid added, and after gentle boiling for a few minutes small pieces of potassium nitrate were then occasionally added, the boiling being made more brisk; the oxidation was complete in twenty to thirty minutes. The nitric acid and other products of distillation condense on the walls of the flask and flow back to the bottom, thus cleansing the sides of the digestion flask. By this method one secures the oxidizing action of the nitric acid at a very high temperature, something like 300° C.

Mr. Wiley presented that portion of the report on dairy products which related to the analysis of butter.

ABSTRACTS OF DATA RELATING TO COMPOSITION AND ANALYSES OF BUTTER COLLATED SINCE LAST MEETING OF THE ASSOCIATION.

THE QUANTITY OF VOLATILE ACIDS PRESENT IN BUTTER FAT.

By Dr. P. Vieth.

[The Analyst, Volume 14, No. 160, page 147.]

In this paper Dr. Vieth gives the results of a great many examinations of butters from different sources, including butter from France, Sweden, Germany, and London; also samples of butter made in the laboratory were examined for content of volatile acids. There is also a description of the effects produced upon the composition of butter fat by different kinds of feed. The results of these studies show that neither barley, wheat, nor oats has an unfavorable effect upon the composition of butter fat. On the contrary, it was seen that the volatile acids decreased when no cereal was given and increased after oats had entered into the diet. It was found that cows in advanced stages of lactation produced a butter in which the content of volatile acids was extremely low, in one case the content having fallen to 14.7 cubic centimeters of deci-normal alkali. Five Jersey cows four weeks after calving produced a butter which contained only 20.3 cubic centimeters of volatile acids.

A METHOD FOR THE ANALYSIS OF BUTTER, OLEOMARGARINE, ETC.

By H. N. Morse and W. M. Burton.

[American Chemical Journal, Vol. 10, No. 4, p. 322.]

The method depends upon the fact that the relative quantities of alkali required to neutralize the soluble and insoluble acids are for any one fat or oil quite constant, but for different fats or oils quite variable. The following statement contains the relative percentages of alkali required to neutralize the soluble and insoluble acids of butter, cocoanut oil, cotton-seed oil, oleomargarine, lard, and beef tallow:

	KOH required for insoluble acids.	KOH required for soluble acids.
	Per cent.	*Per cent.*
1. Butter	86.57	13.17
2. Cocoanut oil (unwashed)	91.95	8.17
3. Cocoanut oil (washed with hot water)	92.43	7.42
4. Cocoanut oil (washed with dilute Na_2CO_3)	92.33	7.45
5. Cotton-seed oil	92.05	7.76
6. Oleomargarine	95.40	4.57
7. Lard	95.96	3.82
8. Beef tallow	96.72	3.40

The mean percentages of hydrate of potash required for the soluble and insoluble acids of butter were found to be as follows:

	Per cent.
KOH for insoluble acids	86.57
KOH for soluble acids	13.17

For cocoanut oil, washed with hot water, the following per cents. were found:

	Per cent.
For the insoluble acids, mean	92.43
For the soluble acids	7.04

For cotton-seed oil the mean percentage of KOH required for the insoluble acid was found to be 92.05, and for the soluble acid, 7.76.

For oleomargarine the mean percentage of KOH required for the insoluble acids, 95.40; for the soluble acids, 4.57.

For lard the mean percentage of KOH required for the insoluble acids, 95.96; for the soluble acids, 3.82.

For beef tallow, mean percentage of potassium hydrate required for insoluble acids, 96.72; for soluble acids, 3.40.

THE REAGENTS.

1. A solution of hydrochloric acids of such strength that 1 cubic centimeter of it is equivalent to 20 milligrams of potassium hydroxide.
2. A solution of hydrochloric acid one-tenth as s'rong as the first.
3. A solution of potassium hydroxide in 95 per cent. alcohol, approximately equivalent to the first acid.
4. A solution of potassium hydroxide in 95 per cent. alcohol, one-tenth as strong as the preceding.

THE PROCESS.

Between one and two grams of the fat is placed in an Erlenmeyer flask and treated with an amount of alkali which is found to be equivalent to 40 cubic centimeters of acid No. 1. The flask is then placed upon the water-bath and heated to the boiling point of alcohol for twenty minutes. The excess of alkali is determined by acid No. 1, using phenolphthalein as indicator. The flask is then returned to the water-bath and heated until all the alcohol has been expelled.

During the evaporation of the alcohol the soap, which in the first place was neutral, becomes alkaline, doubtless owing to the evaporation of a small quantity of volatile acids in the form of esters. After the removal of the alcohol the soap is treated with a quantity of acid No. 1 exactly necessary to liberate all the acids contained in it. The flask is again returned to the water-bath and heated until the liquid becomes clear, the flask being heated with a long, glass tube, 400 millimeters in length and 5 millimeters in diameter, to prevent a loss of volatile acids. The contents of the flask are filtered through double thick paper which has been wet with hot water, and then washed with boiling water until the volume of the filtrate equals a liter, care being taken to wash thoroughly the flask itself. The contents of the condensing arrangement are washed into the filtrate. The funnel containing the washed insoluble acids is placed in the flask in which the saponification was effected, the paper pierced, and the contents washed through with hot 50 per cent. alcohol. The soluble and insoluble acids are thus separated, and the amount of alcohol required to neutralize each is next determined.

BUTTER ANALYSIS.

[Nilson. Zeit. Anal. Chem., 1889, Vol. 2, page 177.]

In a flask of 200 cubic centimeters capacity weigh exactly 2.5 grams of fat. Saponify with 5 cubic centimeters of alcoholic potash (20 grams KOH in 100 cubic centimeters, 70 per cent. alcohol). The alcohol is evaporated, the last trace being removed by a current of air. The soap is dissolved in 50 cubic centimeters distilled water, and a few pieces of pumice stone added. Add 20 cubic centimeters of solution of orthophosphoric acid containing 20 per cent. P_2O_5 (specific gravity at 15° 1.125). Connect the flask with a condenser and distil at the rate of 1 cubic centimeter per minute until 50 cubic centimeters distillate are collected.

The distillate is poured into a water-soaked filter 55 millimeters in diameter. The measuring flask and filter are then washed with 50 cubic centimeters hot distilled water and the filtrate made to reach a volume of 100 cubic centimeters. Add a few drops phenolphthalein solution and titrate with deci-normal potash. The volatile acids in the fat are not all obtained by this method.

A second distillation and even a fifth gave notable quantities of acid in the distillate. The following numbers show the respective quantities measured in deci-normal alkali obtained by the successive distillations:

	Cubic centimeters.
1	14.35
2	1.40
3	.60
4	.38
5	.27

It is possible that it is the laurinic acid present which makes it so difficult to get a complete separation between the volatile and non-volatile acids.

The phosphoric is preferred to sulphuric acid for the following reasons: It is certain that no volatile acids can be formed by the action of the phosphoric acid on the glycerine and the fatty acids during the process of distillation, as is possible when sulphuric acid is used.

By the use of phosphoric acid, therefore, the formation of volatile acid decomposition products is wholly avoided, and this, rather than the danger of the sulphuric acid being carried over in the distillate, is the chief reason for preferring the former.

WOLLNY'S METHOD OF BUTTER FAT ANALYSIS.

Sendtner (seventh report of the Bavarian Chemists' Union, page 92)* has subjected the Wollny method to a thorough examination. He is forced to the conclusion that Wollny has overestimated the possibilities of loss from the following sources:

1. Absorption of carbonic acid.
2. Formation of ethers with sulphuric acid.
3. Coherence of the fatty acids during distillation.

Attention is called to the fact that Goldmann's idea of obtaining all the volatile acids by repeated distillations was clearly foreshadowed by Meissl's statement that the total volatile acid was not recovered in 110 cubic centimeters of the distillate as Hehner and Reichert supposed.

Mansfield's method of avoiding the use of alcohol by substituting an aqeous alkali solution for saponification was also previously used by Legler. The objection to this method is the difficulty of securing complete saponification.

In regard to the removal of the last traces of alcohol by an air blast, it is important that the time of this operation be as short as possible. The blast should also be made with a blowing apparatus, and not from the lungs, on account of the large amount of carbonic acid in the expired air. It is important also that the reagents used be absolutely pure, or as nearly so as possible. The alkali employed should be recrystallized from alcohol, and not only be free from nitrates and chlorides but also from carbonates. The soap after the alcohol is driven off should not be left standing, but immediately treated with the water of solution, and the whole process be carried on to completion as rapidiy as possible. In regard to the losses by the formation of ethers, Wollny has stated that this loss may amount to as much as 8 per cent. In order to determine how great this loss was, by the Meissl method, several experiments were carried through. In one set of experiments, the flask during saponification was heated with a reflux condenser until the fat was completely saponified, while with the other set of experiments the saponification was carried on in the flask without a reflux condenser, according to the Meissl method. The greatest difference observed in the two series of experiments amounted only to .38 cubic centimeter of .1 normal alkali, which was equivalent to 1.5 per cent. of the butter fat used. The danger of loss therefore from the formation of ethers is not so great as Wollny has imagined.

---

* See also Chemisches Central-Blatt, 1889, page 238.

Wollny also observed an error amounting to 30 per cent., due to the coherence of the fatty acids. This source of error also was found not to exist in the experiments made. Comparative determinations were made by adding the sulphuric acid and distilling immediately without waiting for the fat acids to melt. The fatty acids were even added before the soap was entirely dissolved in the water. In all these cases the results differed so slightly as to show that the loss due to the coherence of fatty acids does not exist in fact, or at least has been greatly magnified.

In regard to the form of the apparatus and size thereof and the rate of distillation, these were found to have but little influence on the amount of volatile acid, with the exception of the length of the condenser. With short condensers there was a notable deficiency of volatile acids; and all condensers should be at least half a meter long, or longer. The following method, which is essentially the one proposed by Meissl, was used in the comparison with the Wollny method:

Five grams of the clear, filtered, and well-mixed butter fat were weighed into a round-bottomed flask of 300 to 350 cubic centimeters capacity, and placed upon the water-bath. To the melted fat are added by means of a pipette 10 cubic centimeters of alcoholic potash ley (20 grams potash in 100 cubic centimeters alcohol). The flask is well-shaken in order to mix the potash ley with the fat, and the alcohol mostly escapes in the form of vapor. After about seven minutes, the mass begins to foam, and the saponification is speedily finished. By means of the blowing-bag at intervals of about one-half a minute, a blast of air is passed into the flask, which is constantly shaken, and this is continued until the odor of alcohol disappears. This requires from 15 to, at most, 25 minutes from the time of the commencement of the saponification until the alcohol is entirely removed. One hundred cubic centimeters of distilled water are now added to the soap, the flask lightly covered and placed upon the water-bath until the soap is entirely dissolved. At a temperature of 50° are added 40 cubic centimeters of dilute sulphuric acid (1 to 10) and a few pieces of pumice stone. The flask is immediately connected with the condenser, and the distillate collected in a clean flask holding 110 cubic centimeters. The time of distillation may vary from thirty to seventy-five minutes. The subsequent operations are conducted in the usual way. The whole time need not employ more than two and a half hours.

As regards the possibility of detecting an admixture of oleomargarine in butter, the method is far from accurate. In a mixture made of known proportions of butter fat and oleomargarine, the following numbers were obtained by the Meissl and Wollny methods:

Mixture.	Butter fat.	Calculated by Meissl method.	Calculated by Wollny method.
	Grams.	*Grams.*	*Grams.*
1	1.500	1.524	1.228
2	1.921	2.931	2.794
3	2.112	2.931	2.571

From the above numbers it is seen that the greatest variations may occur even in artificial mixtures, the previous value of the components of which has been determined. It will be seen from the above how useless it is to hope to estimate accurately the amount of oleomargarine in a butter fat by any method resting solely on the estimation on the volatile acids.

Mr. Raumer, of Erlangen (*op. cit.*, page 103), contributes some further criticisms of the Wollny modification of the Reichert-Meissl method of estimating volatile acids in butter fat. He also gives analyses showing the percentage of iodine absorbed, the amount of oleic acid calculated therefrom, and the milligrams of caustic potash required for the saponification of 1 gram of the butter fat, and the melting point of the butter fat. These numbers are found on page 103 of the work cited.

In regard to the Wollny method, Raumer * draws the same conclusions as those obtained by Sendtner. He finds that Wollny has very much magnified the errors mentioned above, and that the method as proposed by Meissl is essentially as accurate as that proposed by Wollny. He finds, moreover, that the observations made by others are quite true, that the apparent percentage of volatile acids is very much increased by long heating of the soap in an open dish or even in a flask. He ascribes this fact to a progressive decomposition of the fatty rather than an absorption of the carbonic acid. For his remarks on this subject reference is made to pages 105, 106, 107, 108, and 109 of the work cited. As a result of his investigations he says that contrary to Wollny's theory, carbonic acid plays a very unimportant rôle in the analysis of butter fats, and that the results secured by Wollny are as easily secured by the method of Meissl without the complicated operations required for Wollny's method. He concludes therefore that a careful exclusion of carbonic acid as described by Wollny is entirely unnecessary, and that in addition to this the expectation of obtaining the whole of the volatile acids by repeated distillations is illusory on account of the continued decomposition of the fatty acids present.

METHOD OF DUBERNARD FOR THE EXAMINATION OF BUTTERS.

Note of Dr. Giuseppe Sartori, assistant to the experimental dariy station of Lodi (Italy), February 6, 1889.

[Le Stazioni Sperimentali Agrarie Italiane, February, 1889, page 138 *et seq.*

To the abridged and popular methods for knowing the mixtures of butter and margarine may be added the method of Dubernard, published in the French Practical Journal of Agriculture, May 17, 1888, and afterwards reported by many Italian agricultural newspapers. The process is as follows:

"In a tube of a rather wide diameter 3 grams of butter are introduced, then the tube is immersed in boiling water to a temperature from 95° to 100° C. When the butter is completely melted pour in the tube 5 cubic centimeters of ammonia, which indicates 30 to the alcoholmeter (density of 0.96). Shake the mixture and put again the tube in boiling water. The ammonia under the influence of the heat volatilizes and escapes, producing a foam more or less consistent, and which rises to various heights in the tube, according to the proportions of margarine which are in the butter submitted to examination. If the butter is pure, large bubbles are formed which burst at the surface."

Having been convinced of the value of phenomena indicated by Dubernard, very visible when the products are pure, I proceeded to experiment with that method and determine its sensibility with mixtures. I secured several samples of margarine butter very well adapted to this experiment, together with samples of pure butter and margarine which entered into the preparation of the mixtures. By means of the determination of the volatile acids with the Wollny method on the samples of the original materials as well as on those of the mixtures, I established the proportions of butter and margarine, which formed these mixtures. Here are the results of my observations:

(1) Butter: volatile acids 26.62 cubic centimeters; large bubbles, few in number, bursting immediately at the surface of the liquid.

(2) Margarine: volatile acids 0.33 cubic centimeters, little bubbles, compact, very white, persistent, which rise in the whole length of the tube. Three strata or layers are noticed: superior, foamy; median, soapy and white; inferior, milky.

(3) First mixture: butter 89.52 per cent., margarine 10.48 per cent. volatile acids; 23.87 cubic centimeters. Compared to No. 1, it was impossible to notice any differential mark or character.

---

* *Op. cit.* and Chem. zeit. Repertorium, 1889, page 41.

(4) Second mixture: butter 82.84 per cent., margarine 17.16 per cent., volatile acids 22.22 cubic centimeters. The froth is more persistent than No. 1; differential characteristics not marked.

(5) Third mixture: butter 71.66, margarine 28.34, volatile acids 19.17 cubic centimeters. Characteristics more sensible; notable difference in the quality of foam, which is whiter, finer, and more compact than No. 1, and somewhat persistent.

(6) Fourth mixture: butter 60.86, margarine 39.14, volatile acids 16.83 cubic centimeters. The characters met with in No. 5 are here much exalted.

(7) Fifth mixture: butter 51.38, margarine 48.62, volatile acids 15.53 cubic centimeters. The foam becomes whiter, finer, and more persistent, and rises up to the middle of the tube.

N. B.—In the Nos. 3, 4, 5, 6, 7 the bottom of the tube is stratified like No. 1.

These experiments have convinced me that with the method Dubernard, a mixture of butter and margarine, this last being not less than 25.30 per cent., can be distinguished; below 25 per cent. the different characteristics can not be established safely. The test with ammonia of Dubernard is good, but unfortunately the weak side of this method is that some butters of low value in volatile acids appear with it as if they were formed only of margarine or are strongly margarine-like. This is a grave defect.

The conclusion is that the ammoniacal test has to be prudently used and only as a supplementary means of testing the butter, being liable to erroneous indications as to the nature of the butter subjected to analysis.

CONTRIBUTION TO THE STUDY OF THE FATTY VOLATILE ACIDS OF BUTTER.

By Prof. PELLEGRINO SPALLANZANI.

[Le Stazioni Sperimentali Agrarie Italiane March, 1889, pages 277 *et seq.*]

In the programme of the work of the zootechnical and dairy school of Reggio, of the Emilian province, was comprised the problem to which this article is devoted.

We propose to establish the fact of variability in the fat of the butter in its proportions of volatile acids, to decide the question of the limits of variation, and to investigate some of the causes to which they are due. We have added to the school a dairy, in which milk is worked all the year round, and we are sure that all the samples upon which we experimented were given to us by faithful and worthy persons, and we believe we have the certitude of the absolute genuineness of the butter subjected to our experiments.

We had at our disposal five breeds of cows—the Rheggian, Swiss, Simmenthal, and Dutch. Each animal was almost equally fed, and the milk of each breed was set aside and the butter was prepared as usual. Each breed had young and old cows, some fresh in milk, and others which were advanced in lactation. The work was conducted in all cases alike. Butters thus obtained were analyzed to determine if the contents of volatile acid varied from breed to breed. The determination of the acids was made by the Reichert method, improved according to the directions of Meissl and Wollny, without neglecting the most minute precautions in taking the sample, in the preparation and use of the agents, in the adjustment of the apparatus, and in the operations in order to secure successful results.

The number of cubic centimeters of decinormal solution of soda which were required to saturate the volatile acids of 5 grams of fat varied between a minimum of 20.63 and a maximum of 30.60. As the analyzed products were surely pure, and the analysis for all was rigorously conducted on the same method, we can affirm that the titre in volatile fat acids of the butter is variable, and that the limits of variations are represented by the quantities which for saturation require 20.63 to 30.60 cubic centimeters of decinormal solutions of soda.

The process of Wollny can discover the sophistication of the butter if the oleomargarine mixture amounts to 30 per cent., but for smaller quantities it can probably, but not certainly, lead to the discovery. As for the influence of the locality upon the quality of the butter, a comparison of the results obtained led to the following conclusions:

Only one among the seven butters from a high altitude shows a value inferior to 24, while two out of four from a low altitude were below that number, and the average of the values for butters of hills or hilly regions was of 24.96, while the average of others scarcely reached 24.03.

All this shows that, proportions of food and care being equal, the butters of high regions without distinction of breeds are better than those of low regions. Besides, also, the cows at the beginning of milking give a better quality of butter than those of an advanced lactation.

The following general conclusions are drawn:

(1) The titre in volatile fat acids of the fat of buttter varies between widely separated limits.

(2) For the butters of the Reggian province, and perhaps for those of the frontiers adjoining, minimum and maximum of volatile acids for 5 grams of butter are represented by 20.63 and 30.60 cubic centimeters of decinormal solution of soda.

(3) Probable causes of the increase of volatile acids are the higher altitude of the site of origin of the butter, the precocity of the bovine race when a rich alimentation is joined to that altitude, and the increase of the fatty globules of the milk.

(4) On the contrary, the diminution is determined most probably by the impoverishment of the milky secretion due to old age of the cow, by the rancidity of the butter, if the butter is kept in the ordinary manner, the washing being done after a certain interval from the time of its manufacture or fabrication.

APPLICATION OF THE METHODS REICHERT, MEISSL, AND WOLLNY TO MIXED BUTTERS.

Note of Prof. Charles Besana, director of the Dairy Experimental Station of Lodi.

[Le Slazione sperimentale Agrarie Italiane, 14th July, 1888, pp. 526, *et seq.*

I subjected to the Woolny method a few samples of commercial artificial butter. The results of the examination are as follows:

Samples.	Professor Wollny's method, cubic centimeters of decinormal alkali.	Point of fusion.
		°C.
Sample of margarine of Milanese manufacture, averages of three concordant analyses	0. 30	42
Sample margarine prepared by author	0. 19	47
Olive oil	0. 14	....
Sesame oil	0. 15	....
Arachis oil	0. 10	....
Butter of cocoanut	7. 94	25. 1
Artificial or margarine butter	0. 99	37. 4
Artificial butter with 90 per cent of margarine	4. 50	36 8
Artificial butter having 75 per cent. of margarine.	7. 26	36. 5
Artificial butter having 90 per cent. of margarine.	3. 44	38. 2
Artificial butter having 75 per cent. of margarine.	6. 41	37. 5

In any mixed butter resulting from the mixture of natural butter with another fatty matter its titre by Wollny's test will be determined by the quantity of pure butter which enters in the mixture, which can be calculated after knowing the three terms, viz, the titre of the mixture, that of the butter, and that of the other fat.

We call b the quantity of butter per 100 (unknown).
m the quantity of foreign fat per 100 (unknown).
t the titre of the mixture, Wollny's test, expressed in cubic centimeters of decinormal alkali.
t' the titre of the butter used.
t'' the titre of the foreign fat.

From the above are derived the two following equations:

$$(1)\quad 100 = b + m$$

$$(2)\quad t = \frac{bt'}{100} + \frac{mtt''}{100}$$

From which the value of b is

$$(3)\quad b = \frac{100\,(t - t'')}{t' - t''}$$

Substituting for t' the numeral values of t' and t'', the per cent. of pure butter existing in the mixture can be known. To verify if the theory was conformable to experiment I prepared mixtures of butter of a known titre with margarine and subjected them to the Wollny test.

The next table contains the data obtained.

Margarine in the prepared mixture.	Butter with titre 27.80 cubic centimeters.	Test Wollny; titre found.	Butter per 100, calculated according to formula.	Difference between the weighed and the calculated butter.
Per cent.	*Per cent.*		*Per cent.*	*Per cent.*
8. 37	91. 63	25. 00	89. 81	−1. 82
16. 67	83. 33	23. 79	85. 40	−2. 07
23. 34	66. 66	18. 95	67. 81	−1. 15
41. 67	58. 33	16. 53	59. 01	−0. 68
50. 00	50. 00	13. 86	49. 30	−0. 70
58. 33	41. 67	11. 70	41. 45	−0. 22
75. 33	25. 00	7. 30	25. 45	+0. 45
83. 33	16. 67	4. 65	15. 81	−0. 86
91. 63	8. 37	2. 60	8. 36	−0. 01

Thus evidently experiment can not furnish mathematical precision. There are errors of observation and of perhaps unknown cause; however, the approximate estimate which we obtain is such that it can practically satisfy anyone.

I have only confirmed by my own experiments those of other experts. Reichert and Meissl have adopted two constant numbers for the titre of butters and fat, and they propose the following formula:

$$B = a\,(n - b)$$

in which B is the quantity of butter per cent. (100), n the titre found in cubic centimeters, and a and b are two constant numbers. That formula is derived evidently from the same reasoning I have used. If in my own formula t' and t'' are admitted as constant figures, viz,

$$t' = 28.8$$
$$t'' = 0.3$$

the formula becomes simplified and reduced to that of Reichert. At the present time margarine is the basis of artificial butters, and if vegetable oils enter into the mixture the difference of the titre is of no importance. Therefore, without falling into grave error, we can concede a constant titre to foreign fats; but it is different with the natural butter. The constancy of the titre does not exist, as has been demonstrated by all the analyses made by many experts.

As a starting point I take, for instance, the figures found by me on the 114 genuine Italian butters. The extremes are 21.8 and 30.2 in round numbers, and I retain now 0.3 for the titre of margarine.

About 85 per cent. of the butters examined by me give a titre comprised between 27 and 30.2 cubic centimeters.

Since we are unable to know the titre of the butter used for the preparation of the mixture under examination, we calculate three values of b by substituting successively in the formula

$$b=\frac{100\,(t-t'')}{t'-t''}$$

the numbers 21.8, 30.2, 27.

Here is a numerical example. Let us suppose we have found by the Wollny test for the titre of a mixed butter 10 cubic centimeters. The three powers of b that I have from the expressions

$$\frac{100\,(10-0.3)}{21.8-0.3}\qquad\frac{100\,(10-0.3)}{30.2-0.3}\qquad\frac{100\,(10-0.3)}{27.0-0.3}$$

are, respectively, 45.11, 32.44, 36.32.

Therefore it may be warranted that that mixture shall contain from 32.44 to 45 per 100 of natural butter.

FROM THE AGRICULTURAL EXPERIMENT STATIONS OF ITALY.

[Vol. XVI, No. V, pages 699 to 702, inclusive. Rome, May, 1889.]

Report of Prof. C. Besana upon Question V, proposed by the Congress of Italian Agricultural Chemists.

"What are the methods of analysis by which we are able to distinguish with certainty and possibly with facility natural butter from artificial butter and their mixtures?"

In November, 1887, our congress announced as the most valuable method for distinguishing natural butter from the artificial butter and their mixtures that based upon the employment of the volatile fatty acids, and also advised the Dronot method, reserving the right to continue successive experiments upon the same.

At the same time the writer was directed to study the butters of the different regions of Italy for the purpose of establishing the limits of the affinity between the quantities of fixed and volatile acids and to ascertain whether the above-mentioned method was in general use and successful in its application.

I will give a brief history of this question from that period to the present time.

A few months after our deliberations there was also formed in Germany a commission, composed of chemists and technicists, who formulated a programme for the collecting of results from the best methods in use in the analysis of butter and their relative fundamental principles, and they invited chemists of Europe and America to take part in this work.

We are pleased to have preceded, to some extent, the German commission in the initiation of these studies which have the final design of guaranteeing the efficient application of the law regarding substitutes for butter, and precluding the possibility of making certain gross errors in judging of the purity of butter, which, now and then appearing in print, cause distrust on the part of the public and give discredit to chemistry, causing the belief that it is incompetent to solve the question of which we are treating.

From November to the present time very few new methods have come to my knowledge.

I will cite the method of Mr. V. Lookeren Campagne and that of Mr. Dubernard, both empirical methods (the second much better than the first), which leave much to be desired, according to the experiments which I have made, although it may be said that some progress had been made with regard to the discovery of easy methods.

Various societies have given out prizes for competition to encourage the discovery of easy methods in distinguishing artificial and mixed butters from the natural butter, but have not had satisfactory results up to the present time.

Certainly the attention of chemists has been much more concentrated upon the method of the determination of the volatile acids.

First, Dr. Wollny, of Kiel (in 1887), then Mansfield, Goldmann, Sendtner, Raumer, and Longi, made this the object of experiment and discussion.

The modifications of the Reichert-Meissl method which were suggested by Wollny, although of little importance, seemed to me so reasonable that I adopt them in the examination of Italian butters.

A method in which there was formerly little confidence, namely, the determination of the specific gravity of butter at 100° C., has been notably improved by Dr. Ambühl, through the construction of an areometer provided with a thermometer, the former having a scale of 0.856 to 0.876. This, also, as an experimental method, seems to me useful, if we employ a bath of salt water in heating the butter to 100° C.

Meanwhile the question here treated has been studied also with great interest and diligence in Italy. Important works have been published upon these questions, and nearly all of them have appeared in the periodical "Le Stazioni Sperimentale Agrarie Italiane," and from foreign periodicals we learn that the works published by Italian chemists are held in high esteem.

The cheese station at Lodi assiduously followed the order given.

The writer published in 1888 the result of the application of the Reichert-Meissl-Wollny method upon 114 specimens of genuine Italian butter, and afterwards a pamphlet upon the methods adopted to distinguish artificial butter from natural butter and their mixtures, in which was made a critical and partly experimental examination of the methods proposed by different authors up to 1888, and concluded by proposing the "densimetric" method, and of estimating volatile acids, according to Wollny as determinative.

The fact demonstrated for the first time in the Italian butters by the writer, that they were subject to notable oscillations as to the quantity of volatile acids (greater than had been believed) awakened some surprise, but my results were amply confirmed by the successive experiments of Dr. Longi at Rome, of Professor Spallanzani at Reggio-Emilia, of Dr. Vigna at Asti, and of Meissen and Rossi at Modena.

The assistants of the cheese station, Rava and Sartori, published studies, the former upon the Dronot method, the latter upon the Mansfield and Dubernard methods.

Salvatori, of the agricultural station at Turin, published studies upon the Dronot method and upon the determination of the fatty acids in butter.

Meissen and Rossi, from the agricultural station at Modena, published the results of the Wollny experiment upon twenty specimens, nearly all from Modena.

Vigna, of the station (enological, of Asti), published the results of experiments upon twenty-three samples of natural butter and eleven of artificial or mixed butter with the methods, Dronot, the densimetric, and Wollny.

Professor Spallanzani submitted seventy samples of butter to the Wollny method, which were in great part manufactured in the zootechnical and cheese school at Reggio-Emilia.

Doctor Longi, in the pamphlet "Experiments and examinations upon the question of butters," published his studies upon the Reichert-Meissel method, and upon the modifications proposed by Wollny, and the results of experiments upon twenty-six samples of butter manufactured in Rome or in the surrounding country.

The value of the method of determining the volatile acids by means of distillation has been confirmed, and up to the present day has not been surpassed by other methods. Certainly this method is not easy, as the public has supposed, but while always aspiring to discover a more practical method, we do not renounce the use of that which is at present valid and reliable and capable of checking the adulterations of butter.

But it is not sufficient to indicate a method, we have also to show its normal or analogical principles. And this is the graver side of the question and that which requires the application of the method to many kinds of genuine butter collected under varying conditions in order to establish the maximum and minimum of the volatile acids.

The data resulting from the labor of the Italian experimenters will be seen in the following table.

Experiments.	Samples.	Location.	Cubic centimeters $\frac{1}{10}$ normal alkali for 5 grams of fat, Wollny method.	
			Minimum.	Maximum.
C. Besana (Lodi)	114	All parts of Italy	21.80	30.19
G. Sartori (Lodi)	52	Lodi, from cheese station	23.59	30.79
A. Vigna (Asti)	23	Mostly from Astigiano	20.68	31.79
P. Spallanzani (Reggio-Emilia).	70	School at Reggio-Emilia	20.63	30.60
A. Longi (Rome)	26	Rome	22.55	28.40
Meissen and Rossi (Modena).	20	Mostly from Modena	21.56	26.40

In the total of 305 samples of butter were found the extremes 20.63 and 31.79 cubic centimeters, but there were two specimens of rancid washed butter in which Professor Spallanzani found the conditions of 16.20 cubic centimeters and 14.31 cubic centimeters.

I believe these figures to be sufficient to show the limits of oscillation in the volatile acids in Italian butters and to establish the minimum conditions under or below which butter is to be considered not genuine. In order to be sure of the operations of the method in experimenting with butters of unknown origin in commerce, I procured samples from three cities in Lombardy, purchased from the various retail dealers, that is, the pork and milk venders.

From one city I had 46 samples of butter, among which 5 showed data comprised between 1.92 and 4.20 cubic centimeters, and the others showed from 22.15 to 29.97 cubic centimeters tenth normal alkali. From the second city I had 16 samples of butter, which showed from 22.91 to 26.87 cubic centimeters. From the third city I had 24 samples, which showed from 22.35 to 26.86 cubic centimeters.

The first 5 samples are therefore artificial butters and the others are held to be genuine. The densimetric and Dronot methods also confirm this result.

We have therefore not found any figures in the results to cause us perplexity in our judgment.

The Congress referring to the report of Professor Besana, responds to question 5 as follows:

1. The preferable method of analysis for distinguishing with certainty and ease the natural butter from the artificial and their mixtures, is the employment of the volatile acids after the Reichert-Meissel method, following the suggestions of Wollny in the particulars.

2. From the examinations recently made in Italy upon butter it results that, following this method, we can not consider butter to be genuine which shows a value inferior to 20 cubic centimeters of deci-normal alkali for 5 grams.

3. As experimental methods, that is, not decisive, the determination of the specific gravity of the butter at 100° C. and the simple Dronot methods are advised.

EXAMINATION OF BUTTER.

METHOD OF DRONOT.

His method rests upon the fact that genuine butter slowly melted gives a clear liquid, at the bottom of which the water, mixed with the casein, settles. Artificial butter remains turbid and loses this aspect only on heating far above the melting point.

According to Salvatori, pure natural fats of all kinds, oleomargarines, and, usually, natural butter give a very clear fluid on melting. Natural butter is moreover characterized very exactly by the precipitate. In artificial butter the precipitate is amorphous and renders the melted liquid turbid, because it settles much slower than in natural butter. Some samples of natural butter give a turbid appearance after melting as above, and this turbidity depends upon finely divided or emulsified drops of water. In all cases in mixtures of natural and artificial butter, the method is not wholly reliable. (Salvatori Italian Experimental Station Bulletin, vol. 14, 1888, p. 516-526.)

ABNORMAL BUTTERS FROM IRELAND, DENMARK, AND SWEDEN.*

Two samples of Danish butter were analyzed and pronounced adulterated on the following numbers:

	No. 1.	No. 2.
Insoluble fatty acid .... per cent..	89.45	88.59
Soluble fatty acid .... do....	4.20	4.01
Specific gravity at 99°	865.70	866.00

A sample of Swedish butter was also pronounced adulterated which had the following composition:

Insoluble fatty acid .... per cent.	89.80
Soluble fatty acid .... do....	3.40
Specific gravity at 100°	865.50

These butters were claimed to be genuine; it seemed, however, that Denmark and Sweden could not possess a special breed of cows or grow special food which would give the butter such a composition.

One can not help being struck with this phenomenal breed of Danish and Swedish cows, which, according to the information obtained by Mr. Allen, secrete one week perfectly good butter-fat, and without any notice and without change of conditions another week produce what an experience of all British butter would lead us to call a mixture. In the samples of butter reported by Mr. Allen it will be evident from the foregoing remarks that fraud might have been committed. I do not assert it was so committed. I do say, however, that the agent of the Danish agriculturists in England has done the farmer a serious injustice in not completely proving the impossibility of fraud.

In conclusion, the inference, I think, which may be fairly drawn from the foregoing remarks is that the specific gravity method, either of Dr. Bell or myself, and the Angell-Hehner insoluble and soluble fatty acid determination, as modified by Dr. Muter and others, are still the most reliable methods of determining the genuineness of butter. Following is a table showing the analyses of the Danish, Irish, and Swedish butters by different analysts.

---

* Estcourt, the Analyst, Vol. 14, page 51.

Collected details of so-called abnormal butters.

	Specific gravity.	Insol. fatty acids.	Sol. fatty acids.	Reichart.
SOMERSET HOUSE.	*At* 100° *F.*			
Danish butter, 1884:				
1	(*)	89.35	4.18	....
2	(*)	88.85	4.24	....
Fresh butter, 1885	....	....	....	....
Danish butter, 1885	908.42	90.08	3.48	....
Swedish butter, 1888, Wigan, No. 402	909.4	90,00	3.32	....
Swedish butter:				
G, 1888	....	....	....	....
N, 1888	....	....	....	....
Danish butter:				
B, 1888	909.9	90.00	4.50	....
O, 1888				
ESTCOURT.				
Danish butter, 1884:	*At* 210° *F.*			
1	865.70	89.45	4.20	....
2	866.00	88 59	4.31	....
Fresh butter, 1885	865.10	92.07	....	....
Danish butter, 1885	862.50	90.12	3.50	....
	At 212°			
Swedish butter, 1888, Wigan, No. 402	865.5	89.80	3·40	....
Swedish butter:				5 *grams.*
G, 1888	864.5	89.7	3.53	23.60
N, 1888	864.0	89.0	3.57	22.55
Danish butter:				
B, 1888	(†)	90.2	4.51	22.44
O, 1888	866.30	89.8	4.70	24.64
ALLEN.				2.5 *grams butter.*
Danish butter, 1884:				
1	....	....	....	....
2	....	....	....	....
Fresh butter, 1885	....	....	....	7.60
Danish butter, 1885	....	....	....	8.9
PROFESSOR STEIN.				5 *grams.*
Swedish butter, 1888, Wigan, No. 402	....	....	....	24.0
Swedish butter:				
G, 1888	866.0	....	....	24.2
N, 1888	866.0	....	....	23.9
ALLEN.				
Danish butter:				
B, 1888	864.0	90.43	4.44	22.39
O, 1888	864.10	90.10	4.67	24.70

* Not given.
† Not enough of the sample.

VARIATIONS OF VOLATILE ACIDS IN BUTTER FAT.

MENOZZI.

[Chemisches Central-Blatt, third series, volume 19, No. 40, page, 1313.]

Menozzi has found in the analysis of 62 samples of butter no such great variations as have been described by Besana. In the experiments conducted by him the proportion of volatile acid in 5 grams of the butter fat varied between 27.1 and 30.2 cubic centimeters. Experiments were also made by adding compounds of the volatile acids with glycerine to margarine and other fats. Considerable quantities of the volatile acids were found in the distillate when such compounds were used.

ESTIMATION OF THE SOLUBLE ACIDS IN BUTTER FAT.

By V. PLANCHON.

[Moniteur Scientifique, fourth series, volume 2, page 1096.]

The method of determination employed was as follows: re-agents—(1) a half normal sulphuric acid; (2) alcoholic solution of soda ley, of which 25 cubic centimeters correspond exactly to 50 cubic centimeters of the half-normal sulphuric acid; (3) an aqueous soda ley one-fifth normal. For the saponification, flasks of 150 cubic centimeters capacity with wide necks are used. Before these flasks are used, 150 cubic centimeters of 15 per cent. alcohol are heated in them on a water bath to 50° to 55°, and the height to which the liquid rises in the neck at that temperature marked. The saponification is carried on with 5 grams of the pure butter fat heated with 25 cubic centimeters of the alcoholic soda ley for 20 minutes with a reflux condenser. After cooling, 60 cubic centimeters of water are added, and a few drops of phenolphthalein. With the semi-normal acid the titration is now carried on until the liquid is colorless, and then a sufficient additional quantity of acid added until the whole quantity amounts to exactly 25 cubic centimeters of potash ley used for saponification. The whole is now heated upon the water bath to 55° until the insoluble fatty acids are separated in a molten condition. With constant shaking, warm water is added until the liquid rises to the mark on the flask. The flask is then closed, vigorously shaken, allowed to cool, filtered, and in 100 cubic centimeters of the filtrate the acidity determined by the fifth-normal soda solution. The soluble acids are calculated as butyric acid. The insoluble acids may be estimated in the ordinary way in the same sample. The amounts of butyric and insoluble acids obtained on several samples are as follows:

Number of sample.	Soluble acid.	Insoluble acid.
	Per cent.	*Per cent.*
1. Pure butter	4.06	87.80
2. Pure butter	3.85	87.62
3. Pure butter	4.41	87.92
4. Margarine	.21	95.76
5. Margarine	.16	95.60

DETERMINATION OF TOTAL VOLATILE ACIDS IN BUTTER.

By F. GOLDMANN.

[Chemiker Zeitung, August 29, 1888, page 1143.]

Goldmann has continued his experiments in the determination of the total volatile acid in butter fats by distillation in a current of steam. However, after six hours of this distillation he has not been able to remove the whole of the volatile acid. A table is given of the total value of volatile acid measured in cubic centimeters decinormal hydrate solution from twenty different samples of butter. The highest number obtained was 43.20, and the lowest number 34.98. As has been shown, however, by other investigators, there is probably a continued decomposition of the fat, so that the distillation might be carried on indefinitely and the above numbers greatly increased. By a similar method of continuous distillation Goldmann has also found that instead of there being only from .40 to .45 cubic centimeters volatile acid in margarine that this quantity is doubled, amounting to about .90 cubic centimeters.

REPORT ON THE RESULTS OF THE EXAMINATION OF SAMPLES OF BUTTER FAT AND SUBSTANCES USED IN ADULTERATING IT.

The samples were purchased in open market in Washington, D. C., and were entered as follows under their trade names.

No. 6370 (sample No. 1)	Oleomargarine.
No. 6371 (sample No. 2)	Butterine.
No. 6372 (sample No. 3)	Butter.

The samples were carefully melted, filtered through a hot filter, placed in appropriat-bottles and shipped to the members of the Association. Accompanying these same ples was the following letter of instructions:

"In order to secure a full co-operation of the members of the Association of Official Agricultural Chemists in the examination of samples of butter fat it has been deemed best to limit the analytical work to the most essential parts. You are therefore earnestly requested to determine in each of the samples sent you herewith the following: (1) Melting point; (2) specific gravity; (3) volatile acids.

"The samples are numbered 1, 2, and 3, respectively, and have the following constitution: No. 1, oleomargarine; No. 2, butterine; No. 3, pure butter fat.

"The melting point is to be determined by the method described in Bulletin No. 19 of the Chemical Division, Department of Agriculture, page 72, or Bulletin No. 13, part 4, page 439.

"The specific gravity is to be ascertained by weighing in a flask at the boiling point of water (see Bulletin No. 13, part 4, pages 428, 429 and 430).

"The volatile acids are to be estimated by the method described in Bulletin No. 13, part 4, pages 459 and 460.

"Instead of using a closed vessel for the saponification, a flask with a long glass tube or condenser can be employed. Deci-normal soda or potash may also be used instead of baryta.

"The acids used for setting the deci-normal alkaline solutions should be carefully standardized and all burettes and pipettes calibrated. Three separate portions of the acid should be weighed from a weighing bottle and the insoluble salts determined.

"Three blank distillations should be made under same conditions as for the volatile acid determinations except that no fat be used.

"The results obtained are to be entered on the inclosed blanks and returned to the undersigned if possible not later than July 15, 1889.

"Each one receiving a package of the samples is requested to make the analyses, since it is of the greatest importance to have as many as possible participate in the work.

"When results are sent in they should be accompanied by full laboratory notes and any suggestions which may seem profitable."

The set of samples retained in the laboratory was examined with every possible precaution with the following results:

STANDARDIZATION OF APPROXIMATELY SEMI-NORMAL SOLUTION OF HYDROCHLORIC ACID.

Specific gravity of the acid at 25° 1.01068

Percentage of chlorine in triplicate.

	Weight acid taken.	Weight AgCl.	Per cent. Cl.
1	2.0567	.1489	1.792
2	2.0051	.1460	1.801
3	1.9781	.1431	1.789
Mean			1.794
Theory			1.756
Theoretical percentage Cl in AgCl			24.74

STANDARDIZATION OF APPROXIMATELY SEMI-NORMAL SULPHURIC ACID.

Specific gravity of the acid solution 1.01540

Percentage of SO_3 in triplicate.

	Weight acid taken.	Weight $BaSO_4$.	Per cent. SO_3.
1	5.0597	.2916	1.978
2	4.9957	.2880	1.979
3	5.0748	.2928	1.981
Mean........	....	....	1.979
Theory......	....	....	1.970
Theoretical percentage of SO_3 in $BaSO_4$			34.33

Having thus determined the actual content of Cl and SO_3 in the respective acids, the factors to be used in correcting the numbers of deci-normal alkali solution as determined by the respective acids are easily calculated. The factor for the solution set by HCl is 1.756 ÷ 1.794=97.9. The factor for the solution set by H_2SO_4 is 1.970 ÷ 1.979=99.5.

STANDARDIZATION OF APPROXIMATELY DECI-NORMAL ALKALI IN TRIPLICATE.

1	10 cubic centimeters H_2SO_4=48.92	cubic centimeters alkali.
2	10 cubic centimeters H_2SO_4=48.88	cubic centimeters alkali.
3	10 cubic centimeters H_2SO_4=49.05	cubic centimeters alkali.
Mean................................	48.95	
1	10 cubic centimeters HC1=49.57	cubic centimeters alkali.
2	10 cubic centimeters HC1=49.71	cubic centimeters alkali.
3	10 cubic centimeters HC1=49.78	cubic centimeters alkali.
Mean................................	49.69	

The approximately deci-normal barium hydrate solution was measured from an accurately calibrated burette at the same temperature, viz, 25° to 27°, at which all the determinations were made. The value of this solution as compared with an accurately measured and weighed 10 cubic centimeters of the hydrochloric acid was 49.69 cubic centimeters of the barium hydrate solution. Since the hydrochloric acid employed was stronger than semi-normal we find the strength of the barium hydrate solution reduced to strictly deci-normal value to be as follows: 49.69 × 97.7=48.65 instead of 50.

As standardized by the sulphuric acid employed we have the following data: For 10 cubic centimeters of the accurately measured and weighed sulphuric acid we require 48.95 of the barium hydrate; then 48.95 × 99.5=48.70 cubic centimeters instead of 50. The barium hydrate solution therefore employed was stronger than semi-normal in the proportion of 48.68 cubic centimeters to 50 cubic centimeters.

In order, therefore, to reduce the results obtained to a strictly deci-normal barium hydrate standard these must be multiplied by 50 ÷ 48.68=1.027.

The blank determinations of the volatile acid in the reagents employed, gave the following numbers:

Blank.	Cubic centimeters, $\frac{N}{10}$ BaO_2H_2.
No. 1.	.20
No. 2...............	.30
Mean...........	.25

In sample No. 1, for 5 grams of fat, the following data were obtained:

	Cubic centimeters, approximately, $\frac{N}{10}$ BaO_2H_2.
No. 1	1.73
No. 2	1.94
Mean	1.84

Then $1.84 \times 1.027 = 1.89$, and $1.89 - .25 = 1.64$ = volatile acid in 5 grams of sample No. 1 measured by $\frac{N}{10}$ BaO_2H_2 solution.

Volatile acid in Sample No. 2, butterine.

	Cubic centimeters, approximately, $\frac{N}{10}$ BaO_2H_2.
No. 1	2.73
No. 2	2.72
Mean	2.73

Then $2.73 \times 1.027 = 2.80$, and $2.80 - .25 = 2.55$ = volatile acid in 5 grams, sample No. 2, expressed in cubic centimeters, approximately, deci-normal BaO_2H_2.

Volatile acid in Sample No. 3, pure butter.

	Cubic centimeters, approximately, deci-normal, BaO_2H_2.
No. 1	27.34
No. 2	27.07
Mean	27.22

Then $27.22 \times 1.027 = 27.95$, and $27.95 - .25 = 27.70$ = volatile acid in 5 grams butter fat measured in cubic centimeters, approximately, deci-normal BaO_2H_2.

SUMMARY OF VOLATILE ACIDS IN SAMPLES.

Volatile acid expressed in duplicate in cubic centimeters deci-normal barium hydrate solution in 5 grams of fat.

Number.	1.	2.	Mean.	Corrected mean.
1	1.78	1.99	1.89	1.64
2	2.80	2.79	2.80	2.55
3	28.08	27.80	27.94	27.69

DETERMINATION OF THE SPECIFIC GRAVITY.

The determination of the specific gravity of the samples of melted fat was carried on in picnometer flasks holding about 25 cubic centimeters of pure water. The comparison of specific gravity is made directly with equal volumes of water and fat at the temperature of boiling water, which in this locality averages about 99°.5. The picnometer is filled with recently boiled distilled water which is still near the boiling point in order to avoid any absorption of air. An example of the method in which the analytical data were obtained is given as follows:

Sample of butter No. 1, first determination.

	Grams.
Weight of picnometer, No. 8	9.9154
Weight of picnometer, No. 8, + water at boiling point	38.1704
Weight of water at boiling point	28.2550
Weight of picnometer, No. 8, + fat at 99°.5	35.2226
Weight of fat	25.3072

Specific gravity=25.3072÷28.2550=.89568.

The data obtained in the three samples are as follows:

Number.	1.	2.	Mean.
1	.8957	.8958	.89575
2	.8943	.8957	.89490
3	.8990	.8999	.89945

MELTING POINT.

The melting point of the samples was determined in triplicate and the following results were obtained:

Number.	1.	2.	3.	Mean.
1	28.25	28.55	28.25	28.35
2	34.25	34.35	34.25	34.28
3	33.15	33.05	33.00	33.07

ANALYTICAL NOTES.

Weighing the acids.—In weighing the acids for the standard determination a tared beaker was weighed with a watch-glass cover. After the acid to be weighed was placed in the beaker the watch glass prevented any evaporation during the weighing. The beaker containing the acid was made perfectly clean, thoroughly wiped with a silk handkerchief, and allowed to stand fifteen minutes in the balance without desiccating material before weighing. After the addition of the acid it was weighed immediately, the beaker being handled in such a way as to avoid the deposition of any foreign substance.

Weighing the fats.—The flasks in which the saponifications were to be made were thoroughly cleaned, dried, and wiped with a silk handkerchief and allowed to remain in a tray prepared for holding them, and covered by a silk handkerchief for fifteen minutes before the weighing began. After the flasks were weighed approximately 5.5 cubic centimeters of the filtered and dried fat were run into each flask from a warm pipette. After the fat in the flasks had cooled to about the temperature of the balance the flasks were again weighed and the amount of fat taken noted. In the weighings which I made the flasks used were counterbalanced by a slightly lighter flask of the same size suspended from the weight arm of the balance. Since, however, it is the increase in the weight of the flask rather than the absolute weight which is desired, this precaution is not necessary to secure accurately the weight of the fat taken. As in the case of the beaker glasses above mentioned, the flasks were weighed without any desiccating material in the balance.

The saponification of the fats.—The saponification of the fats was carried on in strong, oblong round bottom flasks made especially for this purpose and of about 250 cubic centimeters capacity. After the fat is weighed into the flask 10 cubic centimeters of 95 per cent. alcohol are added. The saponifying material, consisting of 2 cubic centimeters of a solution of caustic soda (100 grams to 100 grams of water) kept free from carbonic acid, is next added. A soft cork is then placed in the flask and tied down by a piece of twine. The flask is then placed on the steam bath and allowed to remain, with occasional gentle shaking, for an hour. After the flasks are cooled to about room temperature the stoppers are removed.

Preparation of the acids for distillation.—The soap obtained by the above process was dissolved in 100 cubic centimeters of recently boiled distilled water and to the soap solution were added 40 cubic centimeters of dilute sulphuric acid containing 25 cubic centimeters of the acid to the liter. In order to facilitate the melting of the fat acids, which are separated in the form of an emulsion, the stoppers were replaced in the flasks and they were again placed upon the steam bath until complete fusion of the fatty acids took place. This required different times for different samples. In some instances where butter contains vegetable oils, or where it is made from the milk of cows fed on cotton-seed meal, the time required for the decomposition of the fat acid emulsion may be several hours. This emulsion we have shown to consist of the fat acid and sulphate of soda and water. It is absolutely necessary that the emulsion be decomposed and the acid melted to a clear layer before distillation begins. After the fat acid is melted the flask is cooled to room temperature and the stopper removed.

The distillation.—A few pieces of pumice stone thrown white-hot into distilled water and kept therein are placed in the flask, which is connected by a bulb bent tube with an appropriate condensing apparatus. The flask is supported by a clamp around the neck and is heated by a naked flame carefully and slowly until the boiling point of its contents is reached. The lamp is then so adjusted as to distil 110 cubic centimeters in as nearly as possible thirty minutes.

Titration of the volatile acids.—The 110 cubic centimeters of the distillate obtained as above is filtered through a dry filter until 100 cubic centimeters have passed through. To this 100 cubic centimenters in a beaker glass .5 cubic centimeter of phenolphthalein solution is added and the titration carried on with deci-normal barium hydrate until a red color is produced. The contents of the beaker are then poured into the measuring flask to remove the last traces of acid and the titration continued until the red color remains permanent for a few minutes.

Determination of the melting point.—The melting points were determined with the apparatus described in Bulletin No. 19, and very few additional notes need be made of the method. Great care should be taken that the temperature of the bath be raised gradually and uniformly, the water of the bath being constantly stirred with the blowing apparatus employed. Another precaution should be noticed in regard to the preparation of the disks, in order that no bubbles of air adhere to them. The distilled water which is used in connection with the lump of ice on which the disks are formed should be recently boiled so that all bubbles of air are removed. If the alcohol and water contain air-bubbles, or if the disks have particles of air adhering to them, the work of determining the melting point is unsatisfactory. The first determination in each series should be for the purpose simply of fixing approximately the temperature at which the disk melts. This can then be used as a guide in regulating the temperature of the bath, this temperature being so regulated as not to exceed at its maximum by more than 1° or 1.5° the melting point of the disk. The large test tubes holding the alcohol-water mixture should be cooled in a long beaker filled with water and pounded ice, and the temperature of this cooled mixture will be sufficient to lower the temperature of both to the proper point for the next determination.

The movement of the thermometer used in stirring should be gentle but somewhat constant, so that there should be no marked difference between the temperature of the film of the alcohol-water touching the sides of the tube and the temperature of the interior thereof. In all cases extreme care must be observed not to allow the edge of the disk to touch the sides of the tube. With proper precautions it does not seem possible to get discordant results.

Individual results of analysts.—

LABORATORY NOTES ON BUTTER ANALYSIS.

NEW HAVEN, CONN., *July* 27, 1889.

DEAR SIR: Inclosed I hand you Dr. Osborne's results. The thermometer used was a standard one, graduated to tenths and calibrated by the Yale observatory. The burettes were graduated to tenths and calibrated in duplicate by different persons.

Very truly yours,

E. H. JENKINS.

Dr. H. W. WILEY,
U. S. Department of Agriculture, Washington, D. C.

LINCOLN, NEBR., *July* 9, 1889.

DEAR SIR: Inclosed please find blanks filled in with results of my examination of butter samples.

You will observe that I have filled but one of the blanks, "mean and corrected mean," in expression for volatile acids. This is because these blanks arrived after the calculations were all made, and I did not observe the double columns until filling said blank.

Corrections in all cases were made as follows: 110 cubic centimeters were distilled off, 100 cubic centimeters were titrated, and 0.1 added to the result; from this amount was deducted the number of cubic centimeters required to neutralize the blank; finally this result was reduced to a basis of 5 grams of fat:

Amount w'd off.				Cubic centimeters required.		
5.0068	:	5	::	27.08	:	27.04.

Very respectfully,

HUDSON H. NICHOLSON,
Professor Chemistry, University of Nebraska.

Dr. H. W. WILEY,
Washington.

EXPERIMENT STATION,
Fort Collins, Colo., July 2, 1889.

DEAR SIR: I did not have enough to fill a 100 cubic centimeter flask; larger amount would give better results. The elevation of Fort Collins is 5,218 feet, which might alter the specific gravity somewhat. In Nos. 1 and 2 a *white solid* came over when it was distilled, so I had to clean out the condenser each time. The volatile acids determined as Bulletin 13, Part 4, page 460.

Very respectfully,

DAVID O'BRINE.

Dr. H. W. WILEY.

ITHACA, N. Y., *July* 5, 1889.

Specific gravity.—I used plain picnometers with perforated stopper, holding about 10 grams of sample. The small difference between the fictitious and genuine butters does not seem to me enough to be of any value. Four determinations were made on each sample agreeing within .0003. The method is simple, but was bothered at first by air bubbles in the flask. The results as given are not corrected in any way, as I supposed such correction was not desired.

Melting point.—Followed Wiley's method exactly. Used normal $\frac{1}{10}$ degree thermometer.

In other respects the method was followed in every detail. No especial difficulties worthy of notice occurred. The results given are those resulting from the subtraction of cubic centimeters used for blank from cubic centimeters in determination.

W. P. CUTTER.

GENEVA, N. Y., *July* 13, 1889.

MY DEAR SIR: I inclose results for butter analyses, samples Nos. 1, 2, and 3.

My $\frac{N}{10}$ NaOH solution had just been standardized with potassium tetroxalate as received from Dr. Caldwell for fodder analyses and checked with oxalic acid. Pressure of work prevented my restandardizing as indicated. The laboratory methods used are those indicated in circular. Specific Gravity in specific gravity bottle 40° C.

Melting point.—Wiley's method, volatile fatty acid, Reichert-Wollny method.

Trusting the results may conform to those of other workers,

I am, sincerely yours,

E. F. LADD.

Dr. H. W. WILEY,
Washington, D. C.

MADISON, WIS., *July* 12, 1889.

DEAR SIR: The three samples of fat for the Association of Official Agricultural Chemists were received only a few days ago, they having been through carelessness in the direction sent to Washington Territory, and I don't know where else before coming here. They were nearly a month on the road.

I have made the examinations requested and herewith inclose report of my results. I give upon a supplementary sheet the actual weights and quantities taken for each determination. The numbers given for the volatile acids upon the blanks sent are calculated for 5 grams of fat from the actual amounts taken. The specific gravities were taken in an ordinary picnometer at the boiling point of water, which at this place is between 97° and 99° C., Madison being about 1,000 feet above sea-level. The actual temperature of the determination was not taken. No difficulty was met with in this determination, and no suggestions are offered.

The method given for the melting points is also very satisfactory, duplicate results coming very close together. I don't see how it can be improved.

In estimating the volatile fatty acids I had considerable trouble, the source being in the re-agents used. In my first trial I used the ordinary sodium hydrate in sticks, and after waiting twenty-four hours for the carbonates and other salts to settle the solution was still slightly turbid. Blank determinations made with this solution required 1.3 cubic centimeters of one-tenth normal alkali to neutralize 100 cubic centimeters of the distillate, showing that something was decidedly off. A solution was next made from sodium hydrate purified by alcohol. This after standing a few hours was perfectly clear, but blanks made with it required, as a mean of three determinations, .65 cubic centimeter of one-tenth normal alkali. Although this was a great improvement over the first, the amount of alkali required was still nearly three times as much as it should be. I thought then that the sulphuric acid used must be impure, and made a distillation with 40 cubic centimeters of the acid, and 100 cubic centimeters of water, no soda being used. One hundred cubic centimeters of this distillate required less than .1 cubic centimeter of one-tenth normal alkali. My next trial was with 2 cubic centimeters of sodium hydrate, 100 cubic centimeters of water, and 40 cubic centimeters of sulphuric acid. This distillation required .15 cubic centimeter of one-tenth normal alkali. I then thought that my trouble was due to absorption of carbonic acid during the saponification, and while the alkali was being distilled off. To avoid this, safety tubes, containing caustic alkali, were attached to the whole apparatus, so that I did not think it possible for any carbonic acid to reach the contents of the flask. Blanks made in this way required .7 cubic centimeter of one-tenth normal alkali, being no better than where no special care to exclude carbonic acid was taken. It occurred to me then that the alcohol might contain some acetic acid, although it had been carefully redistilled before commencing my work, and had not been thought of as a source of error. The alcohol was again distilled, with the addition of a little caustic soda, and blanks made with the alcohol thus purified required only .15 cubic centimeter of one-tenth normal alkali. I don't know how common this impurity is, but I think a word of caution in this direction may save others considerable trouble. After this obstacle was overcome no other difficulty was met with in the determination.

The apparatus used and method of procedure were essentially as described in Bulletin 13, page 460, the only modification in the apparatus being that the tube connecting the flask with the condenser was slightly contracted at the lower end, so that it would retain a drop of water, and had a hole blowed in the side of the tube about three-fourths of an inch from the end. This opening always remains open during the distillation, and prevents the possibility of a drop of condensed water and any acid which may have spattered on to it being carried over by a sudden evolution of steam.

The contents of the distillation flask were heated slowly, but it is very difficult to melt the fatty acids to a clear liquid without boiling, and this was not always done. I don't know how much difference this will make. In all other respects the operation was performed strictly according to directions.

In standardizing deci-normal alkali the following weights of HCl were taken:

	Weight of HCl taken.	Required of 1-10 alkali.
	Grams.	*Cubic centimeters.*
1	9.8922	47.46
2	9.9024	47.56
3	9.9666	47.81

The amounts of deci-normal alkali for 10 grams given in furnished blank are calculated from above.

The specific gravities were all made in the same flask.

It was weighed empty before being used, and when cleaned after the determination with each fat—four weighings in all. Three weighings were made when filled with water at the boiling point as below.

	Weight of flask empty.	With water at boiling point.
	Grams.	*Grams.*
1	13.6288	23.7100
2	13.6285	23.7095
3	13.6286	23.7089
4	13.6288	....
Mean	13.6287	23.7095

The weights of flask and fat at boiling point, from which specific gravities were calculated, are as follows:

Number.	1.	2.
1..........	22. 6790	22, 6777
2..........	22. 6633	22. 6634
3..........	22. 7130	22. 7134

I have never made determinations by this method before, and may have overlooked some precautions that are necessary. My results with No. 3 seem too low for pure butter, but aside from this I have no reason to suspect that they are wrong. I should have liked to have had more time to give to this matter, but the sample came so late that I was unable to repeat the determinations. At present Professor Henry is away and my assistants are all away on vacations, so that I have to give my attention to many things aside from work of this kind. I trust that the outcome of these examinations may be a method satisfactory to all.

Very truly, yours,

S. M. BABCOCK.

Dr. H. W. WILEY,
Department of Agriculture, Washington, D. C.

The weights of fat taken for estimation of volatile fatty acids and cubic centimeters of deci-normal alkali required, were as follows:

Number.	Weight.	Cubic centimeters alkali.	Weight.	Cubic centimeters alkali.
1..........	5. 0094	. 75	5. 0130	. 8
2..........	5. 0900	1. 4	5. 0037	1. 6
3..........	4. 9343	25. 6	5. 0100	25. 98

The amounts for 5 grams given in furnished blanks are calculated from these.

S. M. BABCOCK.

COLUMBIA, MO., *July* 20, 1889.

DEAR SIR: Since I sent you the results of butter analyses I repeated all the examinations, with the exception of the specific gravity of No. 1. (The bottle which you sent to Dr. Schweitzer arrived broken; the contents of it had to be filtered and some of the fat was lost.)

For the determinations of volatile acids I made two blank distillations for each analysis. The work was carried out with the same care as before and with the additional help of a little more experience. I repeated these analyses for my own information and send you the results, thinking you might wish to use them in place of those which I sent you first or in addition to them.

Respectfully,

B. VON HERFF,
Columbia, Mo.

Dr. H. W. WILEY,
Chemist, Department of Agriculture,
Washington, D. C.

STATE UNIVERSITY OF MISSOURI,
DEPARTMENT OF CHEMISTRY,
COLUMBIA, MO., *July* 12, 1889.

DEAR DOCTOR: The samples and letter arrived here in good time, but we were so torn up by internal difficulties, that I felt obliged to turn over the whole matter to the assistant chemist of the station, who has done the work very faithfully.

P. SCHWEITZER.

Dr. H. W. WILEY,
Department of Agriculture,
Washington, D. C.

SPECIFIC GRAVITY.

The water-bath used consisted of a porcelain casserole of 375 cubic centimeters capacity. The picnometer was held by a wire which kept the flask about one-half centimeter from the bottom of the vessel. The thermometer inserted in the briskly boiling water did not rise above 98°, but if a few drops of fat were swimming on the surface of the water the temperature reached 100°. The specific gravity of the fat was taken at the same temperature at which the flask was graduated. The diameter of the neck of the picnometer was 8 millimeters. A narrow neck would be perferable.

Weight of picnometer empty, 9.7587.

Capacity of picnometer at the temperature of boiling water.

Determinations.	Gl. + water.	Glass.	Capacity.
			In grams.
1	33.9620	9.7587	24.2033
2	33.9587	9.7587	24.2000
3	33.9530	9.7587	24.1943
4	33.9635	9.7587	24.2048
5	33.9612	9.7587	24.2025
6	33.9560	9.7587	24.1973
7	33.9480	9.7587	24.1893
8	33.9520	9.7587	24.1933
Mean of 8 determinations			24.1981

	1.	2.
Sample No. 1	31.4383 9.7587	31.4284 9.7587
	21.6746	21.6697
Sp. gr	0.89571	0.89551
Mean	0.89561	
Sample No. 2	31.4090 9.7587	31.4140 9.7587
	21.6503	21.6553
Sp. gr	0.89471	0.89491
Mean	0.89481	
Sample No. 3	31.5165 9.7587	31.5230 9.7587
	21.7578	21.7643
Sp. gr	0.89915	0.89942
Mean	0.89928	

STRENGTH OF DECINORMAL ACID (HCl.).

Sodium carbonate C. P. was ignited in a platinum crucible, weighed after cooling, emptied in a beaker, and reweighed. Dissolved in water, heated and titrated with the acid, using cochineal, freshly prepared, as indicator. When nearly neutral the liquid was boiled, to expel CO_2, and then the titration completed. The point of neutralization can thus be very accurately determined.

Final test of acid.

	Na_2CO_3 taken.	Acid used.	0.053 Na_2CO_3 require of this acid.
	Grams.	*Cubic centimeters.*	
(1)	8.4152 8.2267 0.1885	35.64	10.02
(2)	10.0744 9.8651 0.2093	39.53	10.01
(3)	12.3074 12.0814 0.2260	42.64	10.00
			Mean 10.01

STANDARDIZATION OF DECINORMAL SODIUM HYDRATE.

Final test (using phenolphtaleine).

	Acid.	Alkali.
(1)	20	20.10
(2)	20	20.12
(3)	20	20.10
Means	....	20.11

10.05 alkali are equivalent to 10.00 Acid.

VOLATILE ACIDS.

After the digestion with soda lye the alcohol was driven off by blowing air into the flask by means of a rubber bulb. This is done in a few minutes, and saves at least one-half hour's time. The condenser was used for subsequent distillations without cleaning it.

Weighing.—5.5 cubic centimeters melted fat were measured and run into the flask. The desired weight was obtained by means of a glass rod. If done in this manner the weighing is very easy.

Test of reagents.

Blanks.	Cubic centimeters. $\frac{N}{10}$ alkali.
(1)	0.85
(2)	0.78
Mean	0.81

Sample No. 1.

		$\frac{N}{10}$ alkali.	$\frac{N}{10}$ alkali.
(1)	6.0870g 1.0822g	Cubic centimeters, 1.11.	
Fat	5.0048g	5 grams require	1.109
(2)	5.0124g 0.0042g	Cubic centimeters, 1.15.	
Fat	5.0082g	5 grams require	1.148
		Mean	1.128
		Correction from reagents	0.81
			0.318
		Correction from decin. alk	0.005
			0.313

Sample No. 2.

		$\frac{N}{10}$ alkali.	$\frac{N}{10}$ alkali.
(1)	6.0963g 1.0822g	Cubic centimeters, 2.09. 5 grams require	2.084
Fat	5.0141g		
(2)	5.0064g 0.0042g	Cubic centimeters, 2.05. 5 grams require	2.049
Fat	5.0022g	Mean	2.066
		Correction from reagents	0.810
			1.256
		Correction for $\frac{1}{10}$ *n* alk	0.005
			1.251

Sample No. 3.

		$\frac{N}{10}$ alkali.	$\frac{N}{10}$ alkali.
(1)	6.0888g 1.0822g	Cubic centimeters, 26.30. 5 grams require	26.265
Fat	5.0066g		
(2)	5.0101g 0.0042g	Cubic centimeters, 26.32. 5 grams require	26.288
Fat	5.0059g	Mean	26.276
		Correction for re-agents	0.810
			25.466
		Correction for $\frac{1}{10}$ *n* alk	0.125
			25.341

NOTE.—It was impracticable for us at the time to determine the strength of one-tenth normal acid by precipitation with silver nitrate; it was carefully done by sodium carbonate. If it is necessary the determinations can yet be made by precipitation.

R. C. KEDZIE.

The melting point of the fats was determined in the manner directed in the bulletin (No. 19), with the exception that it was necessary to use a test tube 2½ centimeters in diameter and 21 centimeters long. Instead of the rubber bulbs a foot-bellows was used for stirring the water, which proved very convenient. The thermometer was graduated to one-fifth of a degree, each degree occupying a space of .5 centimeters which enabled the reading to tenths with tolerable accuracy.

The specific gravity was determined in specific gravity flasks of a capacity of 50 grams at 60° F. These were placed in boiling water contained in an iron dish at a temperature of a fraction of a degree less than 100° C.

Much more difficulty was experienced in obtaining constant results with water than with the fats.

The following are the results of the estimates of the amounts of water at 100° F. which the flasks contained.

	Grams.
No. 1	48.1138 48.1050 48.1175 48.0835 48.0811 48 0952 48.0931
Mean.....	48.0985
No. 2	48.1041 48.1041 48.1006 48.0978 48.0996
Mean.....	48.1014

The contents of each flask were therefore assumed to be 48.100 grams.

Taking the density of water at 100° C. as .95865 (The Analyst, April, 1889, page 67), and at 15°.5 as .99910, and assuming water at 15°.5 as unity, the specific gravity of water at 100° is .959514.

$$.95865 \div .99910 = .959514.$$

The 48.100 grams of water at 100° will then occupy 48.100 ÷ .959514 = 50.1295 cubic centimeters.

Fat No. 1 (Flask No. 1).

a....Weight of fat, 43.0594 ÷ 50.1295 = .85896 specific gravity.
b....Weight of fat, 43.101 ÷ 50.1295 = .85894 specific gravity.

Fat No. 2 (Flask No. 2).

a....Weight of fat, 43.0594 ÷ 50.1295 = .85896 specific gravity.
b....Weight of fat, 43.0584 ÷ 50.1295 = .85894 specific gravity.

Fat No. 3 (Flask No. 2).

a....Weight of fat, 43.2789 ÷ 50.1295 = .86334 specific gravity.
b....Weight of fat, 43.2794 ÷ 50.1295 = .86335 specific gravity.

Reagents for the estimation of the volatile acids were made as directed.

Soda-lye solution was made from E. Merck's natr. caustic in Bacill.

$\frac{N}{10}$ stand. soda solution was used, prepared by means of $\frac{N}{10}$ oxalic acid in pure water and making up to 250 cubic centimeters. Its accuracy tested by $\frac{N}{100}$ solution of potassium tetroxalate.

$K\,H\,C_2O_2H_2C_2O_4+H_2O=253.52$ Caldwell.

Monatomic equivalent = 84.567.

From the solution of $\frac{N}{10}$ NaHO a hydrochloric acid solution was prepared as a basis of comparison.

$$\frac{N}{10}\ \text{NaHO 50 cubic centimeters} = \frac{N}{2}\ \text{HCl 10.}$$

In the $\frac{N}{2}$ HCl solution the Cl was determined by precipitating with $AgNO_3$ as directed in Fresenius, page 428, last edition.

The solution to be estimated was weighed by running 50 cubic centimeters into a small flask, weighing carefully, pouring into another flask and ascertain the loss of weight from the first flask.

The fat for analysis was weighed in a 300 cubic centimeter Erlenmeyer flask, as recommended in Bulletin No. 13, part 4, page 460.

Two cubic centimeters soda-lye and 10 cubic centimeters alcohol were added, the flask connected with a return condenser and placed in a boiling water bath fifteen minutes.

The alcohol was then distilled off in forty-five minutes. During this time a very small stream of air, which had previously passed through strong soda-lye to remove CO_2, was drawn through the flask by means of a filter pump. This stream was continued until the soap had completely dissolved in water. The H_2SO_4, solution and pumice stone were then added; the flask connected with a glass condenser; the insoluble acids melted and the distillation completed. One hundred cubic centimeters of the 110 cubic centimeters distilled off was treated with the $\frac{N}{10}$ NaHO with the precautions directed.

A 30 cubic centimeter Mohr's burette with pinch-cock was used. Graduated space occupies 40 centimeters of tube. Graduated to one-tenth cubic centimeter.

The following are the results in detail:

No. 1.	First.	Second.
Weight of fat .... grams..	5.077	5.056
$\frac{N}{10}$ NaHO consumed .... cubic centimeters..	1.5	1.3
$\frac{N}{10}$ NaHO consumed in blank .... do...	.5	.5
$\frac{N}{10}$ NaHO consumed by acid .... do....	1.0	.8
$\frac{1.00\times 5}{5.077}=.984$ $.984\times 1.10=1.08$ $\frac{.80\times 5}{5.056}=.795$ $.795\times 1.10=.87$		
$\frac{N}{10}$ NaHO for 5 grams fat .... cubic centimeters..	1.08	.87
Time of distilling .... minutes..	33	33

No. 2.	First.	Second.
Weight of fat .... grams..	5.128	5.295
$\frac{N}{10}$ NaHO consumed .... cubic centimeters..	2.4	2.4
$\frac{N}{10}$ NaHO consumed in blank .... do....	.5	.5
$\frac{N}{10}$ NaHO consumed by acid .... do....	1.9	1.9
$\frac{1.9\times 5}{5.128}=1.85$ $1.85\times 1.10=2.03$ $\frac{1.9\times 5}{5.295}=1.79$ $1.79\times 1.10=1.97$		
$\frac{N}{10}$ NaHO for 5 grams of fat .... cubic centimeters..	2.03	1.97
Time of distilling .... minutes	30	....

No. 3.	First.	Second.
Weight of fat ....grams..	5. 069	5. 236
$\frac{N}{10}$ NaHO consumed ....cubic centimeters..	26. 80	27. 7
$\frac{N}{10}$ NaHO consumed by blanks ....do....	.50	.5
$\frac{26.30 \times 5}{5.169} = 25.94$ $25.94 \times 1.10 = 28.53$ $\frac{27.2 \times 5}{5.236} = 25.97$ $25\ 97 \times 1.10 = 28.67$		
$\frac{N}{10}$ NaHO required for 5 grams ....cubic centimeters..	28. 53	28. 67
Time of distilling ....minutes..	28	30

AGRICULTURAL AND MECHANICAL COLLEGE, AUBURN, ALA.

[Notes on butter analysis for Professor Wiley, Chemist, Department of Agriculture.]

Preparation of $\frac{N}{2}$ HCl.—Prepared HCl of specific gravity 1.1. Weighed out 91 grams of this solution and diluted to 1,000 cubic centimeters to make approximate $\frac{N}{2}$ solution. To standardize, weighed out three portions of 10 cubic centimeters each and determined Cl by $AgNO_3$. Results given on inclosed blank.

Ten cubic centimeters of this solution contain 0.189352 grams HCl, or 1,000 cubic centimeters contain 18.9352 grams HCl. To be standard, 1,000 cubic centimeters should contain 18.185 grams HCl. 18.9352 : 18.185 : : 1,000 : 960.38 = number of cubic centimeters which contain 18.185 grams HCl. Hence 960.38 cubic centimeters of the solution diluted to 1,000 cubic centimeters will make the accurate $\frac{N}{2}$ HCl solution.

One hundred cubic centimeters of this $\frac{N}{2}$ diluted to 500 cubic centimeters make the standard $\frac{N}{10}$ HCl solution, used in titrating the soda solution.

Preparation of Standard H_2SO_4.—Twenty-five cubic centimeters H_2SO_4 specific gravity 1.84 is diluted to 1,000 cubic centimeters, and three portions each of 10 cubic centimeters are treated with $BaCl_2$ to determine SO_3. Results given on blank.

Ten cubic centimeters found to contain 0.34931 of SO_3 or 0.4279 grams H_2SO_4. So 42.7905 : 24.5 : : 1000 : 572.55 = number cubic centimeters which contain 24.5 grams H_2SO_4. Dilute 572.55 cubic centimeters to 1,000 cubic centimeters which gives the standard $\frac{N}{2}$ H_2SO_4. Two hundred cubic centimeters of $\frac{N}{2}$ diluted to 1,000 cubic centimeters make the standard $\frac{N}{10}$ H_2SO_4.

Preparation of $\frac{N}{10}$ Soda Solution.—Eight grams of NaHO in use in the laboratory dissolved in 1,000 cubic centimeters H_2O gave the approximate solution. This was titrated with $\frac{N}{10}$ HCl and $\frac{N}{10}$ H_2SO_4 each, with results as per blank.

Melting point and specific gravity.—Determined without variation from the prescribed methods. Volatile acids by Wollny's method.

Used 3 cubic centimeters of 50 per cent. soda solution instead of 2 cubic centimeters as prescribed, and 60 cubic centimeters of H_2SO_4 solution (25 cubic centimeters in 1,000 cubic centimeters H_2O) instead of 40 cubic centimeters of same. From thirty-five to forty-five minutes were required to distil over the 110 cubic centimeters of the liquid; thirty minutes being called for in the method, The first three or four attempts at distillation were unsuccessful, owing to explosion, which were found to be due to violent "bumping." This bumping was afterwards prevented by the use of coils of platinum wire.

Respectfully submitted.

J. T. Anderson,
First Assistant Chemist.

Dr. N. T. Lupton,
Chemist Agricultural Experiment Station, Auburn, Ala.

State of South Carolina,
Department of Agriculture, Office of State Chemist,
Columbia, S. C., July 18, 1889.

Dear Sir: Inclosed please find the analyses of samples Nos. 1, 2, and 3 of butter fats received from you some time since. We expected to have the analyses ready before the 15th instant, but this line of work being new to us we met with one or two back-sets.

First. We found considerable difficulty in determining when to read the thermometer in the determination of the melting points. The globule in 1 and 2 becoming more or less transparent and rounded at the edges, though elongated and of irregular globular shape. We found, then, considerable difficulty in determining the meaning of the language of the Bulletin on this point.

Second. The Bulletin does not direct the use of pumice stone in the final boiling (distillation) for estimation of volatile acids. This is a serious omission, as it necessitated the making over of all our blanks—it being, according to our experience, impossible to boil the mixture without the pumice stone.

Third The differences in our determinations of the volatile acids are much too great in one or two cases, and are inexplicable. We would search for the cause, but fear the delay would make us too late in getting in our results.

We followed the method as directed, but used an Erlenmeyer flask with long glass tube as reflux condenser in the saponification, and after adding the H_2SO_4 solution distilled from the same Erlenmeyer.

Hoping the result will prove satisfactory, I am,

Very respectfully yours,

W. D. Burney.

Dr. H. W. Wiley.

WORK ON BUTTER-SAMPLES.—WALKER BOWMAN.

Determination of specific gravity at 100° *C.*—Determinations made by weighing in a flask at the boiling point of water, as recommended in Department Bulletin No. 13, part 4, pages 428, 429, and 430. Must be borne in mind, however, that our station is situated at an unusual elevation above the sea-level (about 2,500 feet), the average barometer-reading being 705 millimeters, and water boiling at 97.5° C. Thus our specific gravity determinations have not been made strictly at 100° C.

	Grams.
Sample No. 1.	
(*a*) Weight of flask empty	17.3448
Weight of flask + water at 100° C	65.6704
Weight of water	48.3256
Weight of flask with fat at 100° C	60.7401
Weight of fat	43.3953
Specific gravity=43.3953÷48.3256=.8979.	
(*b*) Weight of flask empty	17.3448
Weight of flask+water at 100° C	65.5654
Weight of water	48.2206
Weight of flask with fat at 100° C	60.6618
Weight of fat	43.3170
Specific gravity=43.3170÷48.2206=.8983.	
Sample No. 2.	
(*a*) Weight of flask empty	17.3448
Weight of flask+water at 100° C	65.6566
Weight of water	48.3118
Weight of flask with fat at 100° C	60.5646
Weight of fat	43.2198
Specific gravity=43.2198÷48.3118=.8946.	
(*b*) Weight of flask empty	17.3448
Weight of flask+water at 100° C	65.6145
Weight of water	48.2697
Weight of flask with fat at 100° C	60.6079
Weight of fat	43.2631
Specific gravity=43.2631÷48.2697=.8962.	
Sample No. 3.	
(*a*) Weight of flask empty	17.3448
Weight of flask+water at 100° C	65.6238
Weight of water	48.2790
Weight of flask with fat at 100° C	60.8599
Weight of fat	43.5151
Specific gravity=43.5151÷48.2790=.9013.	
(*b*) Weight of flask empty	17.3448
Weight of flask+water at 100° C	65.7382
Weight of water	48.3934
Weight of flask with fat at 100° C	60.8372
Weight of fat	43.4924
Specific gravity=43.4924÷48.3934=.8987.	

Determination of melting point.—Determination made according to method described in Department Bulletin No. 13, part 4, page 439. Results stated above. The melting points in the case of sample No. 2 seem quite anomalous, notwithstanding the fact that I took every precaution, making likewise a number of determinations, of which all fell within comparatively narrow limits.

Determination of volatile acids.—According to method of Wollny, as described in Department Bulletin No. 13, part 4, page 460. Exactly 5 grams taken for each determination. Erlenmeyer flask with condensers used for saponification. Decinormal soda used instead of baryta for titration.

For each determination of volatile acids a blank was treated in the same way—no fat being used—and the distillations were conducted as far as possible under exactly the same conditions, and side by side.

	Grams.
Sample No. 1.	
(*a*) Weight of flask counterbalanced	14.9092
Weight of flask + fat	19 9092
Weight of fat taken	5.0000
Number cubic centimeters $\frac{N}{10}$ sodium hydrate used for volatile acid	1.2
Number cubic centimeters $\frac{N}{10}$ sodium hydrate used for blank	.75
Corrected number cubic centimeters $\frac{N}{10}$ sodium hydrate used for volatile acid	.45
(*b*) Weight of flask counterbalanced	17.8517
Weight of flask + fat	22.8517
Weight of fat taken	5.0000
Number cubic centimeters $\frac{N}{10}$ sodium hydrate used for volatile acid	1.3
Number cubic centimeters $\frac{N}{10}$ sodium hydrate used for blank	.8
Corrected number cubic centimeters $\frac{N}{10}$ sodium hydrate used for volatile acid	.5
Sample No. 2.	
(*a*) Weight of flask counterbalanced	13.4602
Weight of flask + fat	18.4602
Weight of fat taken	5.0000
Number cubic centimeters $\frac{N}{10}$ NaOH used for volatile acid	5.5
Number cubic centimeters $\frac{N}{10}$ Na OH used for blank	.7
Corrected number cubic centimeters $\frac{N}{10}$ NaOH used for volatile acid	4.8
(*b*) Weight of flask counterbalanced	3.9468
Weight of flask + fat	8.9468
Weight of fat taken	5.0000
Number cubic centimeters $\frac{N}{10}$ NaOH used for volatile acid	5.3
Number cubic centimeters $\frac{N}{10}$ NaOH used for blank	.85
Corrected number cubic centimeters $\frac{N}{10}$ Na OH used for volatile acid	4.45
Sample No. 3.	
(*a*) Weight of flask counterbalanced	14.8690
Weight of flask + fat	19.8690
Weight of fat taken	5.0000
Number cubic centimeters $\frac{N}{10}$ NaOH used for volatile acid	31.7
Number cubic centimeters $\frac{N}{10}$ NaOH used for blank	1.1
Corrected number cubic centimeters $\frac{N}{10}$ NaOH used for volatile acid	30.6
(*b*) Weight of flask counterbalanced	15.4904
Weight of flask + fat	20.4904
Weight of fat taken	5.0000
Number cubic centimeters $\frac{N}{10}$ NaOH used for volatile acid	31.4
Number cubic centimeters $\frac{N}{10}$ NaOH used for blank	1.0
Corrected number cubic centimeters $\frac{N}{10}$ NaOH used for volatile acid	30.4

Determination of strength approximately, decinormal sulphuric acid.

	Grams.
(*a*) Weight of bottle empty	17.9811
Weight of bottle + acid taken	38.0462
Weight of acid taken	20.0651
Weight of barium sulphate found	0.2083
Percentage of SO_3 found	0.35
(*b*) Weight of bottle empty	16.6700
Weight of bottle + acid taken	36.6980
Weight of acid taken	20.0280
Weight of barium sulphate found	0.2131
Percentage of SO_3 found	0.36
(*c*) Weight of bottle empty	18.8073
Weight of bottle + acid taken	38.9147
Weight of acid taken	20.1074
Weight of barium sulphate found	0.2148
Percentage of SO_3 found	0.36

AGRICULTURAL AND MECHANICAL COLLEGE OF TEXAS,
College Station, Tex., August 10, 1889.

DEAR SIR: Herewith find analyses of butter samples, which I hope may be satisfactory. In volatile acids we distilled only 100 cubic centimeters in a graduated flask. The method of melting point was unsatisfactory to us, but we managed to get fairly concordant results. The capillary tube seems to us preferable. The weights of $BaSO_4$ was made by one of us only, the titration being checked by both.

Yours truly,

H. H. HARRINGTON.

DR. H. W. WILEY,
Chemist, Washington, D. C.

KENTUCKY AGRICULTURAL EXPERIMENT STATION,
LEXINGTON, KY., *August* 12, 1889.

MY DEAR SIR: I send you herewith inclosed report of examination of butter samples sent out by you some time ago as reporter of Association of Official Agricultural Chemists.

I am sorry that the report has been delayed so long. It would have been reported sooner had not an accident happened to No. 2 sample.

Finding that you could not furnish us another portion of sample No. 2, we concluded to make the determinations and report them, as you probably can make some use of the results. The mishap happened as follows:

The butter fat, No. 3, in the specific gravity bottle, containing between 8 and 9 grams, was placed beside the flask containing No. 2, and unfortunately when it was emptied it was put in No. 2. Of the mixture there were 95 grams, and we calculated 8 grams of butter fat. From this data I have calculated and substracted the amount of one-tenth normal alkali required to neutralize the volatile acids in the butter fat accidentally put in No. 2 and subtracted it from the results.

As the melting point and specific gravity of No. 2 could not be obtained, I report these results as obtained from the mixture. You will find inclosed laboratory notes.

The melting points were obtained in the manner directed in the official method, and we had no difficulty in the manipulation, save that in No. 2 the disks twice happened to touch the edge of the test tube while the water in the bath was about 36 degrees, and this brought the melting point to about 36.

I have given the per cent. of chlorine by weight, and in weighing we took 10 cubic centimeters, so that the calculations are based upon the calibration of our pipettes.

The saponification was made in an Erlenmeyer flask connected with a return condenser. The fats saponified easily in from fifteen to twenty minutes. It took from three-quarters to an hour to drive off the last traces of alcohol. Glass wool was put in the bulb of the tube connecting the Erlenmeyer flask with the condenser in the distillation.

No difficulty was experienced in the distillation.

Yours, very truly,

M. A. SCOVELL.

DR. H. W. WILEY,
Chemist, Department of Agriculture, Washington, D. C.

Prof. A. M. Peter, first assistant chemist, assisted me in this work, and he should have credit for it. Please credit the work from this station to "Scovell and Peter."

M. A. S.

Determination of volatile acids.

	No. 1.		No. 2.		No. 3.	
Amount of mixed sample taken	....	....	*5.1554	*5.0042	....	....
Weight of substance taken (grams)	5.0238	5.0531	4.7212	4.5828	5.0095	5.1865
Actual number cubic centimeters to neutralize 100 cubic centimeters distillate	....	....	*4.65	*4.95	....	....
Cubic centimeters NaOH to neutralize 100 cubic centimeters of distillate	0.85	1.00	2.39	2.76	26.30	27.05
Number cubic centimeters for 110 cubic centimeters or whole distillate	....	....	*5.12	*5.45	....	....
Cubic centimeters NaOH to neutralize 110 cubic centimeters or whole distillate	0.94	1.10	2.63	3.04	28.93	29.76
Substract from blank experiment	0.10	0.10	0.10	0.10	0.10	0.10
Corrected number cubic centimeters NaOH	0.84	1.00	2.53	2.94	28.83	29.66
Calculated to 5 grams substance	0.836	0.990	2.679	3.229	28.775	28.588
Equivalent in one-tenth normal alkali	0.83	0.98	2.66	3.21	28.59	28.41
Mean	0.91		2.94		28.50	

Figures with asterisk (*) show actual results obtained from the mixture of 87 grams of No. 2 with 8 grams No. 3. Other figures for No. 2 are calculated from these.

The saponification was done in an Erlenmeyer flask with return condenser, and the flask was heated on a boiling water-bath fifteen to thirty minutes as seemed necessary. The distillation was from the same flask, and was accomplished in nearly every instance within thirty minutes. A bulb filled with glass-wool was used to prevent spray from entering the condenser.

THE PENNSYLVANIA STATE COLLEGE,
AGRICULTURAL EXPERIMENT STATION,
74 West River street, Wilkes-Barre, Pa.

DEAR DR. WILEY: After longer delay than I had anticipated I am able to put together the data you desired for your report on butter fats. In addition to what the chart contains, I may say that the acid used in titering back the $\frac{N}{10}$ alkali, was made up from 100 cubic centimeters of the N HCl, which was standardized with the results given in the report.

The boiling point with freshly boiled distilled water varied between 98.6 and 98.8, the mean being 98.66°, and the barometric variation being .124. The thermometer used read to $\frac{1}{10}$° C., and was a fine normal, reading up to 100° C.

For a more exact comparison of specific gravity with previous results, I had a determination made at 40° C. also, with these results:

1=.9061. 2=.9038. 3=.9107.

The analytical details of standardization were performed by Dr. H. B. McDonnell; the remaining portion by Mr. G. L. Holter.

Very truly,

WM. FREAR.

VIRGINIA AGRICULTURAL AND MECHANICAL COLLEGE,
EXPERIMENT STATION,
Blacksburgh, Va.

NOTES ON ANALYSIS OF BUTTER SAMPLES.

For the determination of the melting points, specific gravity, and volatile acids, the methods used were those recommended in your circular.

Melting point.—In the first and second samples the same test-tubes were used, but for the third a fresh test-tube was prepared. No difficulty experienced except the adhering of the disks to the spatula, probably because the latter was not cold enough. In every case the water in beaker stood about one-half higher than the test-tube at the melting point. Nos. 1 and 2 ran together much more rapidly than No. 3.

Specific gravity.—In taking the specific gravity of No. 1 I used a 50 cubic centimeter picnometer; for No. 2, a 25 cubic centimeter one, and a 10 cubic centimeter one for No. 3. Those used in determination of Nos. 2 and 3 were of the make suggested in foot note on page 430 (Bulletin No. 13, Department of Agriculture).

Slight difficulty in wiping off the stopper as some of the contents of capillary tube would be absorbed by the blotting paper, or else the water or fat remaining on top of stopper would be drawn in by suction.

Volatile acids.—In the determinations as well as in the blanks (which were conducted at the same time and under the same conditions), the saponification was carried on in Erlenmeyer flasks with condensers. The sulphuric acid used in the first sample was not of the same bottle as used in Nos. 2 and 3. In every case exactly 5 grams of fat was taken.

I would call your attention to the fact that although the specific gravity determinations were made at the boiling point of water, they were not at 100° C., as water here boils at 97.5° C.

I would suggest also that when samples are sent out a description of the sample should be sent with it, telling how the sample was drawn and prepared, etc.

Very respectfully,

B. F. FINNEY,
Assistant Chemist, Virginia Experiment Station.

Weights in analyses of butter samples, Blacksburg, Va.

[Specific gravity at 97.5° C.]

	No. 1.		No. 2.		No. 3.	
	a.	*b.*	*a.*	*b.*	*a.*	*b.*
Weight flask empty	17.3446	17.3446	11.5158	11.5157	8.5015	8.5015
Weight flask + H_2O	65.6905	65.5754	35.8141	35.8051	18.1880	18.1997
Weight H_2O	48.3459	48.2308	24.2983	24.2894	9.6860	9.6982
Weight flask and fat	60.7453	60.7356	33.3186	33.263	17.2531	17.2355
Weight fat	43.4007	43.3910	21.8028	21.7473	8.7516	8.7340
Specific gravity	.8977	.8996	.8972	.8953	.9034	.9005

Volatile acids.

Weight flask counterbalance	—2.2955	15.4455	12.5762	13.4372	12.5672	15.4571
Weight flask counterbalance + fat	+2.7045	20.4455	17.5762	18.4372	17.5672	20.4571
Weight fat taken	5.0000	5.0000	.5.0000	5.0000	5.0000	5.0000

Standard acids.

	1.	2.	3.
Weight Beaker	16.6	16.6	16.6
Weight Beaker + acid	36.385	36.605	36.555
Weight acid taken	19.785	20.005	19.955
Weight crucible + BaS_4O	7.4769	7.9045	6.7095
Weight crucible	7.2673	7.6937	6.4988
Weight $BaSO_4$	.2088	.2108	.2107

LABORATORY NOTES FROM ARKANSAS EXPERIMENT STATION, FAYETTEVILLE.

CALIBRATION OF BURETTES AND PIPETTES.

Average of two determinations.

	Acid (HCl)	Alkali (NaCH)	Pipettes.
First 10 cubic centimeters	9.9447	10.0260	....
Second 10 cubic centimeters	9.9303	9.9480	1.9760
Third 10 cubic centimeters	9.9583	9.9400	....
Fourth 10 cubic centimeters	9.9725	9.9360	9.9370
Fifth 10 cubic centimeters	....	9.9495	....

When acid was titrated against NaOH the first 10 cubic centimeters were used each time.

Standardization of HCl.

	1	2	3
Weighing tube +	23.1600	22.5390	23.2355
Weighidg tube —	13.1865	12.5040	13.2405
	9.9735	10.0350	9.9950
Crucible +	21.6820	20.0675	23.4820
Crucible —	21.0723	19.4575	22.8725
	.6097	.6100	.6095

.1508 Cl. .1507 Cl. .1507 Cl. Mean .15072.

.15072 ÷ 5.1073 = .295107 equivalent of Cl in 1 cubic centimeter approx. $\frac{1}{10}$ normal alkali.

.1775 ÷ 5 = 3.55 equivalent of Cl in 1 cubic centimeter $\frac{1}{10}$ normal alkali.

.295107 ÷ 3.55 = .8313 percentage of $\frac{1}{10}$ normal alkali in approx. $\frac{1}{10}$ normal alkali.

Titration against standard alkali.

	10 cubic centimeters acid.
1	51.05
2	51.10
3	51.07
Mean	51.073

	2		3	
	a.	*b.*	*a.*	*b.*
Reduced to equivalent of 5 grams	2.706	2.712	35.03	35.13
Reduced to equivalent of $\frac{1}{10}$ normal X .8313	2.249	2.254	29.120	29.203

Specific gravities.

	Water distilled.		Fat No. 2.		Fat No. 3.	
	Pic. 1.	Pic. 2.	Pic. 1.	Pic. 2	Pic. 1.	Pic. 2.
Full	48.4850	51.0635	45.6535	48.1960	45.8400	45.8285
Empty	18.7330	20.7210	18.7330	20.7212	18.7330	18.7330
H_2O	29.7520	30.3425	26.9205	27.4748	27.1070	27.0955
Specific gravity	....	....	.9048	.9054	.9111	.9107

Melting points.

	C°.
No. 2	35.6 35.6 35.7
	35.63
No. 3	33.2 33.2 33.3
	33.23

Blank distillation.

[Two cubic centimeters NaOH (100 grams to 100 cubic centimeters H_2O) + 10 cubic centimeters alcohol evaporated — 100 cubic centimeters H_2O added + small pieces pumice stone + 40 cubic centimeters dilute H_2SO_4— all distilled exactly 110 cubic centimeters 100 titrated. Four determinations.]

	1st = 0.45.	2d = 0.50.	3d = 0.47.	4th = 0.50.
Increased $\frac{1}{10}$	0.495	0.55	0.517	0.55
Reduced X .8313	0.412	0.457	0.4312	0.457

Volatile acid.

	No. 2.		No. 3.	
	a.	*b.*	*a.*	*b.*
Flask + ...	47.6340	52.6255	52.0620	52.1865
Flask — ...	42.5540	47.4550	47.0930	47.1900
	5.0800	5.1710	4.9690	4.9965
Titration ..	2.50	2.55	31.65	31.91
Inc. $\frac{1}{10}$	2.75	2.805	34.815	35.101

Comparison of data.—When it is remembered that this year is the first time that any comparative work has been done to any extent in the examination of butter fats it must be conceded that the results obtained in the above table are gratifying. Some parts of the manipulation were performed for the first time by many of the analysts and the data were necessarily obtained therefore without previous practice in the manipulations required.

In regard to the specific gravities it is noticed that the agreement in the data obtained by the same process is very close. I have made no attempt to reduce the numbers obtained at other temperatures than that of boiling water to standard temperature. Unless the station is at a great elevation, such as the one in Colorado, the temperatures at which water boils are so nearly the same that the slight variation from one station to another or the slight differences in the pressure of the atmosphere from time to time could not be observed in the figures obtained. The slight differences noticed are doubtless due to different methods of manipulation, different degrees of care in cleaning the apparatus and making the weighings, and are not inherent in the method itself. Whether or not the numbers should be expressed for water at the boiling temperature or referred to water at its maximum density of 4° is a matter of small importance. Personally, I should prefer to define the specific gravity of a liquid body as the number expressing the weight of 1 cubic centimeter of that body in grams at the temperature of water at 4°. The convenience, however, in having approximately a fixed temperature easily reached by every one, viz, that of boiling water, is so great that for practical purposes I think the method employed is to be preferred to any other.

In regard to the melting points, with a few exceptions, the agreements are very close. The difficulty of graduating a thermometer at the temperature of melting fats is very great. In general we have to rely upon a thermometer graduated at zero and at the boiling point of water, and assume that it is also correct with the changes made necessary with the graduations above mentioned at temperatures between 30° and 40°. The slight variations noticed in the melting points, with the exception of the underscored numbers, may be largely ascribed to differences in the thermometers employed. In regard to the numbers in brackets I can see that in some cases they could only have been reached by mistakes in the manipulation of the method. This is particularly the case with column No. 1. In column No. 2 in the same way it appears strange that the variations marked by the numbers in brackets should have been obtained. It is easy to see how, by differences in manipulation and differences of thermometers, variations of 1° or even 1°.5 might be noticed in different localities; but it is hard to see, where accurate numbers show the melting point to be about 35, how a melting point of 28 could be obtained without some variation in the method of manipulation or some inexplicable mistake.

With pure butter in column No. 3 the variations are not great and the agreement between the different numbers obtained is very close.

Volatile acids.—The most surprising results are those obtained in the volatile acids of samples Nos. 1 and 2. The differences in the data are at once evident in glancing at the table. In some cases the amount of volatile acid obtained is less than that obtained in the blanks, showing almost no acid at all of a volatile nature in the samples. In other cases the amount of volatile acid reached a considerable quantity. In general it may be said that the data obtained for the volatile acids in samples Nos. 1 and 2 are the most unsatisfactory of all the data secured.

In the case of the pure butter the numbers are fairly satisfactory. A few instances evidently show a lack of care in the manipulation or some radical defect, which of course your reporter is not able to point out. From the testimony obtained the case is made out without a question of doubt that the volatile acids in the sample of butter as determined by the method described correspond to about 28 cubic centimeters of the decinormal alkali solution. As 110 cubic centimeters of distillate were obtained the total volatile acid distilled would be 2.8 cubic centimeters more than that indicated, viz, 30.8 cubic centimeters in all. It is possible that in those cases where more than 30 cubic centimeters of volatile acid were indicated the whole of the distillate might have been titrated instead of a little over 90 per cent. of it.

Nevertheless the data show that the members of this association when working by a given formula and following it out in detail, will be able to obtain results on volatile acids which are fairly comparable and reliable.

Table of results obtained by different analysts in the examination of samples of fat for Association of Official Agricultural Chemists.

Names of analysts.	Specific gravity.			Melting point, C°.			Volatile acids corrected, $\frac{N}{10}$ alkali.		
	1.	2.	3.	1.	2.	3.	1.	2.	3.
Scovell & Peter	.8961	[1].8954	.9003	30.60	[1][37.50]	34.50	.91	[1]2.91	28.51
Wm. Frear	[2][.9038]	[.9027]	[.9103]	29.90	35.60	33.40	[5.50]	3.30	[33.00]
H. H. Harrington	8973	.8974	.9023	[21.83]	33.13	33.46	.94	1.76	28.47
B. F. Finney	.8986	.8962	.9019	30.10	[28.63]	34.23	.65	[3.92]	[30.30]
Lupton and Anderson	.8993	.8974	.9006	29.63	35.40	32.67	1.48	1.11	28.14
R. J. Davidson	.8989	.8978	.9024	27.80	34.60	32.80	1.10	1.71	29.21
J. B. McBryde	.9009	.8986	.9030	27.80	34.80	33.10	.97	1.81	28.25
Walker Bowman	.8981	.8954	.9000	29.70	36.60	33.76	.48	[4.63]	[30.50]
(1) B. v. Herff	.8956	.8948	.8993	30.80	36.20	34.40	[.31]	1.25	25.34
(2) B. v. Herff	.8956	.8947	.8991	30.50	35.70	34.30	.53	1.25	25.69
Robt. C. Kedzie	[3][.8598]	[.8589]	[.8633]	29.00	[37.40]	33.40	.47	1.50	28.10
H. C. White	.8962	.8959	.8991	29.55	35.60	34.30	1.02	[4.70]	29.58
C. B. Collingwood	[4]	[.9051]	[.9109]	[4]	35.63	33.23	[4]	1.81	28.72
H. H. Nicholson	[2][.9058]	[.9047]	[.9106]	29.60	34.40	33.30	1.23	1.44	27.06
David O'Brine	[5][.9032]	[.9023]	[.9118]	31.40	[31.40]	[35.70]	.42	1.32	[24.77]
W. P. Cutter	.8962	.8961	.8993]	30.20	[37.20]	34.50	[.14]	[.07]	27.92
E. F. Ladd	[2][.9052]	[.9054]	[.9115]	29.76	34.30	34.30	1.30	1.15	26.25
C. A. Goessmann	.8966	.8952	.8999	29.53	33.20	33.66	.60	1.45	25.55
S. M. Babcock	.8977	.8962	.9012	29.30	34.90	33.80	.62	1.33	25.79
T. B. Osborne	.8980	.8970	.9011	29.60	35.60	34.30	[.21]	1.34	27.00
H. J. Patterson	.8953	.8949	.9002	29.47	35.80	32.00	.56	2.14	29.34
W. Wipprecht	.8976	.8977	.9023	[21.70]	33.30	33.63	.99	1.92	28.76
H. W. Wiley	.8958	.8949	.8995	28.35	34.28	33.07	1.64	2.55	27.69
Means (omitting numbers in brackets)	.8972	.8967	.9006	29.62	35.17	33.64	.87	1.73	27.65

[1] Sample slightly mixed.
[2] Determined at 40°.
[3] Water at 4°.
[4] Sample lost.
[5] Elevation of station, 5,218 feet.

METHOD OF MANSFELD.

The method of Mansfeld as has already been mentioned consists in a saponification of the butter without the use of alcohol. I have given this method several trials, and am convinced that it has much merit. With certain precautions I think it may be made to take the place entirely of a saponification with alcohol, thus avoiding all possibility of loss from etherification, and at the same time avoid the trouble of the complete removal of the alcohol and the danger of leaving any of it present with the soap.

In order to avoid the hard soap which is formed with caustic soda, it is best to perform the saponification with caustic potash, and using as strong a solution as possible, viz, 100 grams of the pure caustic dissolved in 59 grams of water.

I have, however, secured very good results using the caustic soda solution employed in ordinary saponification. I find it necessary, however, in this saponification to use a stoppered flask, as will be shown by the following data:

RESULTS OF THE EXAMINATION OF BUTTER, BY MANSFELD'S METHOD, IN OPEN FLASKS.

Sample No. 1, cubic centimeters decinormal barium hydrate required	25.9
Sample No. 2, " " " " " "	19.3
Sample No. 3, " " " " " "	17.7

The great irregularity in the amount of volatile acid secured in the above experiment shows that the saponification in the open flasks by the Mansfeld method was not complete.

Taking the same sample of butter, and the saponification in a closed flask, the following results were obtained:

Sample No. 1, cubic centimeters barium hydrate required	29
Sample No. 2, " " " " "	29

The same butter examined in the usual way for volatile acids gave the following results:

Sample No. 1, cubic centimeters decinormal barium hydrate required	28.07
Sample No. 2, " " " " " "	28.19
Sample No. 3, " " " " " "	28.06

The results above are in harmony with those which I have obtained before with this method, showing that by it a slightly greater quantity of volatile acid is indicated than by the ordinary method.

Professor Sartori in "Le Stazioni Sperimentali Agrarie" January 19, 1889, pages 513 and following gives the results of the determination of volatile acids in 52 samples of butter by the Wollny and Mansfeld method. These two sets of determinations agree extremely well together. In some cases there is a slightly larger percentage obtained by Mansfeld's method, while in others a slight increase is obtained by the Wollny method. On the whole, however, the agreements are so perfect, as to indicate that Mansfeld's method with proper precaution may be made to take the place entirely of the Wollny method. For the details of Professor Sartori's paper, I refer to the original publication. It is for this reason that it has seemed well to me to recommend to the association the use of Mansfeld's method in connection with the Wollny method during the coming year.

If the results obtained by other members of the association are similar to those obtained by me and by Professor Sartori, it will probably be found advisable, at our next annual meeting, to dispense with the use of alcohol in the saponification of our butter fats, and rely solely upon the Mansfeld method.

Mr. Jenkins moved that the report as far as read be accepted.

The motion was seconded and carried.

Mr. Jenkins said that while he understood, from what he knew concerning the work of Messrs. Wiley and Harrington and others, that abnormal butters were likely to be met with at times still he had so far experienced no difficulty in deciding as to the genuineness of butter from the results of the determination of the volatile fatty acids and of specific gravity. He had examined several hundred samples of butter taken by the dairy commissioners from stores and boarding houses. He explained the methods of butter analysis practiced by himself and assistants, the results of which had been satisfactory. He wished to ask why Mr. Wiley recommended the careful melting of the fatty acids before distillation? He had always heated them gradually and kept them hot while connected with the condenser for a little time before beginning distillation; he would like to know whether there was in that method a source of error.

Mr. Wiley said that the fatty emulsion entangled some of the volatile acid, and in the beginning of the distillation one gets off a lot of material which has not been equally saturated by the whole volatile acid present. For instance, if one-tenth of the volatile acid is mechanically held in the emulsion it does not appear in the distillate, and in the case of some fatty acids which he had dealt with the whole 100 cubic centimeters might be distilled off without complete melting; he had known them to resist three or four hours of boiling temperature.

Mr. Harrington inquired what advantage was gained by distilling 110 cubic centimeters and filtering it, and titrating 100 cubic centimeters.

Mr. Wiley said that he did not know that much advantage was gained by that method; but it avoided the loss by evaporation from a large surface of filtering paper during the whole process. The old method had been to filter as distilled, drop by drop on filtering paper exposed to a large surface of air. He thought the present method was much better than that of filtering at the time of distillation.

Mr. Scovell said that some one had stated that he never filtered at all if the results were accurate. He would like to ask whether the reporter had had experience in that line?

Mr. Wiley said that he never made any titrations without filtering. Of course if acids with higher melting points were carried over mechanically, they were not volatile acids in the true sense and should be removed by filtration.

Mr. Harrington said that in the case of samples Nos. 1 and 2 he had been troubled with the lauric acid distillation spoken of in the report, and unless the filtering took place as the distillation occured he thought there would be difficulty in measuring the 100 or 110 cubic centimeters as the case might be.

It was moved that the recommendations of the reporter covering the method of butter analysis be adopted as a whole.

The motion was seconded and carried.

On motion of Mr. Gaines the meeting was adjourned.

It was understood that the association was to meet at 7 o'clock for the banquet. At this reunion Mr. Myers presided, having the honorable Assistant Secretary of Agriculture at his right as the guest of honor. Twenty-seven members were present at the dinner.

THIRD DAY—THURSDAY.

The president called the meeting to order and stated that the continuation of Mr. Wiley's report upon dairy products was in order. Immediately prior to the adjournment that portion of the report referring to the analysis of butter and fats had been disposed of. The next subject treated of in the report was the analysis of milk.

Mr. Wiley said that he would like to return for a few moments to the report on butter in order to consider the suggestion made by Mr. Jenkins in regard to the Mansfeld method. He had been in the habit of letting the fat solidify and pouring in the alkali throwing it along the sides of the dish so that before any fat touched the sides of the dish it should be united with the alkali. Mr. Jenkins' method, however, seemed to him, although he had never practiced it, a most reasonable one, making a thorough emulsion of the two before saponification began. He would like to ask whether Mr. Jenkins had ever experienced trouble by that method? If not, he should be in favor of adopting it in place of the recommendation contained in the report as being easier and in every respect better.

Mr. Jenkins said that his work had been limited simply to the examination of butter, butterine, and oleomargarine, and had not been extended to other fats, with which he had had no experience. He had never found difficulty with the emulsion. After three hours in the water bath the decomposition was perfect and he had seen no sign of any undecomposed glycerides. He had never compared this method with the other as he was only required to decide whether an article was pure butter or oleomargarine, and he had found in the market in Connecticut no samples as to the character of which there was any doubt. In court he was only obliged to swear that they were or were not pure butter and the puzzling question between butter and butterine did not arise.

Mr. Wiley said that he would like to incorporate in the report the method of saponification as an alternate method with the Mansfeld process.

It was moved that the desired permission be granted.

The motion was seconded and carried.

Mr. Wiley presented that portion of the report on dairy products which related to the influence of food on the composition of butter, as follows:

INFLUENCE OF FOOD ON THE COMPOSITION OF BUTTER.

By C. J. Von Lookren.

[Milch Zeitung, 1889, page 341.]

The influence of food on butter is found chiefly in the variation in volatile acids and in the melting point. The differences in the melting point are undoubtedly the consequence of the different contents of oleine in the butter.

Butters from the neighborhood of Delft, in Holland, from pasture of clay land near the sea, highly manured with stable manure, had melting points as follows: 33.7, 34.9, 33.9, 33.3, 34.0, 34.5, 34.6, 34.7, 33.2, 33.9, 34.2.

The odor and taste of the butters were both good. Butters from clay pasture lands, not manured; melting points were found to be: 36.9, 36.8, 35.7, 35.5, 36.8.

Butter of good quality from the province of Friesland; melting points were found to be: 35.5, 34.9.

From the above researches it can be concluded that differences in melting points at least partly depend upon the nature of the pasture grass. The question would arise, in what way do these differences come about? According to our present knowledge, they may arise in three different ways:

First. Through the kind of soil; for instance, clay, sand, etc.

Second. Through the position of the soil, whether of a high or low elevation.

Third. Through the method of fertilizing and caring for the pasture.

DO THE VOLATILE ACIDS IN THE FOOD OF MILCH COWS OCCUR IN THE MILK?

By H. Weiske.

[Chemisches Repertorium, December 22, 1888, page 341.]

The opinion is very generally prevalent in agricultural practice that the volatile acids contained in certain cattle foods—as, for instance, ensilage—sometimes go into the milk, and not only thereby influence the odor and taste of the milk, but also make the milk readily turn sour. Professor Soxhlet has, however, shown by exact experiments that the ease with which milk becomes sour in such cases does not depend upon the transfer of the acids contained in the food, but much rather on the circumstances under which the food is given, which cause the stalls in which the animals are kept to be infested with all kinds of germs. These germs, during the milking, fall into the milk and cause the ready fermentation by which the milk becomes sour. In a similar way can be explained the influence upon the odor and taste of the milk from cows which consume this kind of material. In an experiment upon a goat which daily received one gram of butyric acid, it was noticed that the milk obtained from the animal had just as good an odor and taste as it possessed before the butyric acid was added to its food. It is therefore not proven that there is any transfer of organic acids from the food of the animals to the milk.

INFLUENCE OF DIFFERENT FOODS UPON THE MELTING POINT AND CHEMICAL COMPOSITION OF BUTTER.

[Mayer, Die Landwirtschaftlichen Versuchs-Stationen, Vol. 35, page 261.]

The following conclusions are drawn from a long study on the influence of different food materials upon the melting point and composition of butter:

(1) The percentage of volatile acids in butter goes hand in hand with the specific gravity of the butter fat. An increase in one shows also an increase in the other.

(2) The melting point of the butter does not vary with the data above mentioned, since it is presumably more dependent on the percentage of oleine then on that of butyrin, capronin, and allied glycerides.

(3) The percentage of volatile acids in a butter fat and also its specific gravity vary

even for a single cow between wider limits than has formerly been supposed when the animal is subjected to different conditions of experiment.

(4) The percentage of volatile acids in the butter fat is dependent upon the period of lactation and falls in general with the length of that period.

(5) The percentage of volatile acids is also in a high degree dependent upon the food of the animal; beets, meadow grass, and green clover in our experiments producing a higher percentage than hay and this a higher percentage than ensilage grass.

(6) The melting point of butter fat is also dependent upon food. Ensilage grass and hay produce butters with the highest melting point: then follow beets, while exclusive green food, whether in pasture or in stall and whether from grass or clover, gives butters with the lowest melting point.

(7) The solidification point of butter fat rises and falls with its melting point in general.

(8) Pasturage produces upon races of cows which are accustomed thereto a very favorable influence upon the production of milk and also of butter.

INFLUENCE OF FOOD ON THE COMPOSITION OF BUTTER.

[Ladd, report of New York Experimental Station for 1888, page 291.]

Studies upon the composition of feeding stuffs, especially upon the albuminoids and oils, have led to the supposition that in some instances the peculiar properties of food affect the composition of butter and other animal products.

Three foods were selected, viz, corn meal, linseed meal, and wheat bran, to be used in connection with hay, to determine if possible what influence they had on the composition of the butter. The amount of iodine absorbed by the different samples and the viscosity of the soaps produced from the fat showed most plainly the differences produced by different varieties of food. As indicated by the absorption of iodine, it was shown that cows having linseed meal for a food produced a butter which had 3.5 per cent. more of oleine, while animals fed with wheat bran apparently produced a butter with a lower percentage of oleine. The viscosity of the butter made from the linseed meal was greater than that of any other of the foods.

NOTE. Since the iodine number of linseed oil is about 155, it is possible that the use of this food would produce a butter having a high iodine absorption without increasing the amount of oleine contained therein or perhaps even diminish it. The experiments of Mr. Harrington and myself, already cited, show that the reducing agent of cotton-seed oil may pass through the organism of a cow practically unchanged. It may be true, therefore, that some member of the linoleic series present in linseed oil and which give to that substance its high iodine number might pass into the butter with the effect noted above.

INFLUENCE OF FOOD, BREED, AND IDIOSYNCRASY ON THE COMPOSITION OF BUTTER.

One of the fundamental principles of dairying is regard for the influence which the care of the animal, supervision of the milking, separation of the cream, ripening of the cream, churning and washing, have on the quality of butter for table use. These processes also, together with the method of packing, have a notable influence upon the preservation of the butter in a sweet state. The discussion of the above problems, however, is a thing for the practical dairyman rather than the chemist. The chemical composition of butter fat, as influenced by the character of food received by the animal, the race of the animal, and the peculiarities of the animal, has hitherto been little studied from a chemical point of view. To the latter subject I propose to devote the following paper.

Late in February, this year, I received a letter from Prof. H. H. Harrington, chemist of the Experimental Station of Texas, accompanied by two samples of butter which he asked me to examine. The following extract from Professor Harrington's letter will indicate the motive which led him to send the samples:

> "Some work in our laboratory indicates that volatile acids from the cotton-seed butter are much lower than has been generally supposed. I send two samples of butter, one from cotton-seed feed and the other from feed containing no cotton seed. If you can do me the favor of analyzing this butter, I shall send more samples from the same cows on the same feed. We hope in the near future to follow up these analyses with complete analyses of butter from different feeds, feeding two cows on cotton-seed and then changing them to other feed."

The two samples of butter received from Mr. Harrington were entered as follows: Butter from cotton-seed (No. 6316); butter from other feed (No. 6317).

The samples sent by Mr. Harrington were small and a complete analysis could not be made; but the results obtained on the small samples sent are of such interest that I will communicate them at the present time and call attention to the peculiarities noticed. When other samples are received they will be examined and compared with the samples already analyzed.

Results of analyses.

	6316.	6317.
Volatile acids, number cubic centimeters $\frac{N}{10}$ BaO_2H_2 for 5 grams..	21.00	28.50
Percentage of iodine absorbed	33.40	31.89
Melting point	45° C.	34°.2 C.
Reduction of silver by Bechi	Distinct.	None.

The most remarkable points connected with the above analyses are as follows:

(1) The low percentage of volatile acids in butter from cotton-seed.

(2) The phenomenally high melting point of the butter from cotton-seed.

(3) The persistence of the reducing agent of the butter from cotton-seed, as indicated by its action upon nitrate of silver.

The melting point of the butter, as will be seen, is higher than that of pure lard. The particular point to be noticed in this matter is that in butter designed for consumption in southern countries, or produced in southern countries, the mixture of cotton-seed with the feed of cows will tend to raise the melting point of the butter and render it more suitable for consumption in hot climates.

The persistence of the reducing agent is also a matter of interest. It has passed, in the samples examined, through the digestive organism of the cow and has re-appeared in the butter with almost undiminished activity. The selective action of the digestive organs on the different glycerides contained in the food of the animal is also a matter of importance. It would be expected *a priori* that the butter from a cow fed largely on cotton-seed oil would contain more olein and have a lower melting point than if ordinary food were used. On the contrary, it is seen that either the more solid glycerides have been absorbed during the process of digestion or that the olein has undergone some distinct change in the digestive organism by which it has assimilated the qualities of the other glycerides.

From an analytical point of view the results are of great importance, since they show that a butter derived from a cow fed on cotton-seed meal, or on one exerting a fat of unusual quality, might be condemned as adulterated when judged by the amount of volatile acids present. Since cotton-seed meal is destined to be a cattle food of great importance, especially in the southern part of the United States, this is a fact of the greatest interest to analysts.

The observation of Mayer, soon to be mentioned, that the specific gravity of butter fat varies with its content of volatile acids, I have also verified by the determination of the specific gravity of samples of butter fat taken from the milk of the same cows kept on the same food, but taken the following day after the samples mentioned.

The specific gravity for the cotton-meal fed sample was .8929 at 99; for the ordinary fed sample, was .8991 at 99.

Professor Mayer's experiments were made on a single cow, of a North Holland breed. From time to time during the progress of the experiments the original food was used, in order to see what effect the period of lactation would produce. The cow was fed for twelve days on each separate ration before the samples were taken. After two days more another set of samples was taken and then the food changed for a new experiment.

In the butter fat the melting and solidifying points were taken, and the volatile acids determined according to the method of Reichert. The specific gravity was also determined by the Westphal method at 100°.

The rations of the cow were composed of the following materials:

Ration No. 1: 15 kilograms meadow hay and 2 kilograms linseed cake.
Ration No. 2: Siloed grass, *ad libitum*, and 2 kilograms linseed cake.
Ration No. 3: 20 kilograms beets, 8 kilograms hay, and 2 kilograms linseed cake.
Ration No. 4: Pasture grass, *ad libitum*.
Ration No. 5: Chopped clover with 14 per cent. of other grasses *ad libitum*.

The highest melting point observed, viz, 40.5, was from ration No. 1; the lowest, viz, 33.5, from ration No. 5. The highest volatile acids were produced by ration No. 3, and required 33.5 cubic centimeters $\frac{N}{10}$ alkali. The lowest volatile acids, viz, 20 cubic centimeters, were observed with ration No. 2.

The results mentioned above were obtained on the first samples of butter sent by Mr. Harrington, and were published in Agricultural Science for April, 1889, pages 80 *et seq.* Not fully satisfied with the result of a single determination I asked Professor Harrington to send me other samples of butter, which he did on two subsequent occasions. The analyses of the two last sets of samples sent did not fully bear out the results obtained in the first set. This led me to believe that perhaps the influence of the animal was not fully allowed for in Professor Harrington's samples. The two last sets of samples were analyzed with the following results: No. 6347, sample from cow fed on corn meal and wheat bran only; Nos. 6348, 6349, and 6350 from cows fed on 2 pounds cotton-seed meal, 4 pounds cotton-seed, and 16 pounds corn and wheat bran. No 6374 from cow having no cotton-seed in food; Nos. 6375, 6376, and 6378 from cows fed solely on cotton-seed meal.

The analytical data obtained are as follows:

Serial number.	Specific gravity at 100° C.	Volatile acid in cubic centimeters $\frac{N}{10}$ alkali	Melting point.
6347		24.70	35.10
6348	(*).9063	27.50	40.60
6349	.9009	27.70	40.30
6350	.9009	25.30	40.30
6374	.8967	19.95	36.60
6375	.8989	27.20	34.45
6376	.8962	25.80	36.63
6378	.8989	25.40	36.23

* This number is probably too high.

The above data are very perplexing. The conclusions derived from the first set of samples are supported solely from the fact that the cows fed on cotton-seed meal gave butters which, with one exception, had a uniformly higher melting point than ordinary butter. The phenomenally low percentage of volatile acids in 6374 would indicate that the cow furnishing this sample was the same as that furnishing the low volatile acids in the first set of samples. An inquiry directed to Professor Harrington to elucidate this point, however, has not yet been answered.

Another supposition is that in some way the numbers of the samples may have been changed in the analysis, but this is scarcely probable. The importance of a more careful study of this subject led me to institute some feeding experiments of my own in order to unravel if possible the mysteries of the preceding analysis. I accordingly obtained authority from the Secretary of Agriculture to arrange for certain feeding experiments with Professor Alvord, of the Maryland Agricultural Experiment Station. Three cows were selected for these experiments, described by Professor Alvord as follows:

No. 1. Full-bred Jersey.
No. 2. Full-bred Ayrshire.
No. 3. Cross-bred Jersey and Ayrshire.

These cows were kept on ordinary pasturage for ten days, and then the milk from each of the cows for three days was taken for the experiments. All the milk was subjected to the same conditions. It was set in earthern bowls in a refrigerator at 45° to 50° F. and skimmed after twelve hours. The cream was mixed and kept at 55° to 60° until the fourth day after the beginning of the milkings. The cream was then ripened in a room at 68° F. temperature for twenty-four hours. After cooling to 62° F. the cream was churned, the temperature rising from 62° at the beginning of the churning to 65° F. at its close. The time required for each churning was twenty minutes. The three days on which the milk was saved were damp hot days, very unfavorable for making good butter. In all cases the butter was thoroughly washed in cool well water, made into rolls and put in glass jars. One-half of each sample of the first lot was salted at the rate of two-thirds of an ounce of salt to one pound of butter.

After the conclusion of the first set of experiments the cows were gradually changed to a ration of cotton-seed meal, using the commercial variety, such as is used for fertilizing purposes, as no unextracted cotton-seed meal could be obtained at this season of the year. The ration of cotton-seed meal was gradually increased, the cows finally being given all they would eat of it. The following are the facts as to the second lots:

The feeding of cotton-seed meal was commenced on the 25th of July, giving but one pound at a feed at first, but constantly increasing the quantity. The milk saved and used for the second lots of butter was that of the 2d, 3d, and 4th of August. Below is given a table showing the quantity of meal consumed, milk, cream, and butter produced from each cow, and time of churning:

Identity of cows and butter.		Cotton-seed meal consumed on August 1, 2, and 3.	Milk product used for the butter August 2, 3, and 4.		Cream from the milk stated churned.		Butter made from the stated cream August 6.		Time of churning for each lot.	Temperature of cream at starting churn.
Lot.	Breed.									
		Lbs.	*Lbs.*	*Oz.*	*Lbs.*	*Oz.*	*Lbs.*	*Oz.*	*Min.*	*° F.*
1	Jersey	28	32	15	10	8	2	05	13	62
2	Ayrshire	32	37	00	9	12	1	12	22	62
3	Cross-bred Jersey and Ayrshire	29	32	04	9	7	1	07	14	62

During this trial the cows were turned into a small lot with very short pasturage, for exercise and access to running water.

They were fed only the cotton-seed meal, and consumed the quantity stated. At close of trial the Jersey and cross-bred cows were beginning to refuse the meal. The Ayrshire continued to eat all offered, and probably could have been fed 12 pounds a day, but I was afraid to give her over 11 pounds a day, and did that only twice. (She later kept on at 8 and 10 pounds per day, while the others fell to 1 and 2 pounds.)

The analytical data obtained on these samples of butter are found in the following table:

Description of samples.—6467, unsalted butter from pure Jersey cow on pasture, no feed; 6468, fresh butter from pure Ayrshire cow on pasture, no feed; 6469, fresh butter from cross-bred Jersey and Ayrshire cow on pasture, no feed; 6470, salted butter, same as 6467; 6471, salted butter, same as 6468; 6472, salted butter, same as 6469; 6478, butter from pure Jersey cow fed on cotton-seed meal; 6479, butter from pure Ayrshire cow fed on cotton-seed meal; 6480, butter from cross-bred Jersey and Ayrshire fed on cotton-seed meal.

In addition to the above samples I have also included two samples of creamery butter from a large creamery at Attica, Kans.

Eight thousand pounds of milk are received daily at this creamery, all of which comes from native cows feeding on native prairie grass, with the exception of a few Holstein cows kept by one person.

6409, butter from Attica creamery, made May 20, 1889; 6473, made July 20, 1889.

Table of analyses.

Serial number.	Melting point, degrees C.	Per cent. iodine absorbed.	Volatile acids in cubic centimeters $\frac{N}{10}$ BaO^2H^2 for 5 grams.	Specific gravity.	Bechi's reaction.	Millian's reaction.	Fatty acids from the above.	
							Crystallizing point, degrees C.	Per cent. iodine absorption.
6467	34.9	37.7	22.8	.9010	None	None	38.95	38.69
6468	36.3	41.1	22.5	.9005	do	do	39.80	42.50
6469	35.2	38.0	22.1	.9019	do	do	38.55	39.20
6470	35.3	37.9	22.3					
6471	35.6	40.7	22.5					
6472	35.2	38.2	22.3					
6478	38.4	34.9	21.4	.9016	Marked	Marked	41 25	37.96
6479	39.0	36.8	20.8	.9012	do	do	43.30	38.72
6480	38.3	35.2	21.1	.9011	do	do	41.45	37.66
6409	33.3		28.1	.9001				
6473	34.4	29.8	29.3	.9021	Trace	Trace	39.60	31.26

The study of the data in the above table reveals many points of interest. In general the data corroborate the results of the first study of the samples sent by Professor Harrington. The melting points of the butters from cows fed on cotton-seed meal are markedly higher than from the other samples. There is also a markedly diminished content of volatile acids in these butters and a lower iodine absorption power. The latter character is unlike the Harrington sample. Another characteristic phenomenon noticed in the first samples of butter is also here repeated, viz, the persistence of the reducing agent which is present in cotton-seed oil in the butter derived from animals fed thereon. The physiological importance of this phenomenon will be mentioned in another place. The most curious results, however, of these experiments is found in the increase in the butter of the glycerides having a high melting point in other words, the glycerides of the palmitic and stearic series. While further experiment may be necessary to show that there is a uniform diminution of volatile acids in butters from cows fed on cotton-seed meal, the fact is now most clearly established that the melting point of such butters is uniformly higher. In regard to the absorption of iodine by the butters from cotton-seed fed cows the results obtained above are somewhat at variance with those secured by Ladd,[1] who states that butter from cows fed on linseed meal contained 3.5 per cent. more olein than those samples which were obtained from cows fed on bran. This conclusion of Ladd's, however, may not be the true one, since linseed oil has an iodine absorption of about 155 per cent. and this high co-efficient may have had some influence upon the butter as regards iodine absorption. It is possible, therefore, that some of the linoleic glyceride, which has so high an iodine-absorbing power, may have found its way into the butter, thus increasing its iodine absorption.

---

[1] Report of New York Experiment Station for 1888, page 91.

Another important characteristic of the butters examined is seen in their abnormally low content of volatile acids. If we compare the samples from the Maryland station with those from Kansas, we have a very characteristic contrast between abnormal pure butter and normal pure butter. The two samples from Kansas show a percentage of volatile acids which is not usually met with in samples of pure butter. On the other hand the samples from the Maryland station show an abnormally low content of volatile acids. This percentage of volatile acids is indeed so low that these butters would be condemned as spurious if we relied upon the volatile acid test alone. It does not seem so strange, in the light of these facts, that Allen should have found abnormal Danish butters which nevertheless from their history were certainly genuine.

In so far as the breed of the animal is concerned in the above experiments, it does not seem to have greatly influenced the composition of the butter. The low content of volatile acids may therefore be attributed either to the food and pasturage or to the peculiarity of the animals themselves, or to the period of lactation.[1] It would hardly seem probable, however, that three animals taken at random should have exhibited in almost the same degree the abnormal qualities indicated in the composition of the butters above.

The physiological questions which are suggested by the above study are of the utmost consequence. In a paper entitled "Note on the action of digestive fluids on oil," published in The Medical News of July 28, 1888, I called attention to the remarkable influence exerted on a large quantity of oil in the human digestive organs. A pint of oil, presumably sweet oil, but more than likely cotton oil, was administered to the patient for the relief of an obstruction in the gall duct. This oil in passing through the digestive organs was completely decomposed, mostly into fatty acid with some soap, forming an emulsion in the alimentary canal, and being voided in the form of rounded masses of considerable consistence, and mistaken by the patient for gall-stones. This action of the digestive liquids was entirely unexpected, and seems to show that the commonly accepted notion that the fats are acted upon solely by being emulsified and thus absorbed into the circulatory fluids is an erroneous one.

It is the common supposition that the fats have for a physiological function the maintenance of the animal heat of the body and the nutrition and supply of the fatty portions thereof.

The experiments in feeding cows on cotton-seed meal would seem to indicate that the natural glycerides contained in cotton-seed meal do not appear in the butter of the cows fed thereon. If the cotton-seed oil in the food should pass unchanged into the butter we might, it is true, have a lowering of the volatile acids, but this would be accompanied by a great increase in the iodine absorption and a marked lowering in the melting point. It is quite certain that the glycerides of butter which yield on saponification volatile acids are not derived from similar glycerides in the food of the animal. It may be also quite true that none of the glycerides in the butter of the cow is derived from the fat of the food of the animal. It is more than likely that the fat of milk is a direct product of digestion, and is formed conjointly from the carbo-hydrates and the albuminoids in the cow's food. We need not, therefore, be perplexed any longer at the presence of so small a portion of stearine and so large a proportion of the butyric series of glycerides in the fat of milk.

---

[1] The dates of calving of the three cows are as follows: (1) Jersey "Mulberry," February 3, 1889; (2) Ayrshire "Junetta," March 23, 1889; (3) cross-bred "Blackie," April 18, 1889. The experiments having been made in July and August, 1889, the low content of volatile acid could not have been due to an advanced period of lactation.

From the evidence already at hand, I think we would be justified in saying that all the fats in milk are products of digestion and none of them results of simple translation through the digestive organs or fats already present in the food. On the other hand, we have undoubted evidence of the translation of other substances directly from the food of the cow to the butter fat, as is shown in the presence of the aldehyd in cotton oil which reduces silver in the butter of cows fed on this substance. Among many other studies on the influence of the food on the composition of butter I might cite the paper of Ladd, already noted, and also one by C. J. Von Lookeren published in the Milch Zeitung, No. 3, 1889, page 47; and the paper of Mayer published in Die Landwirtschaftlichen Versuchs-Stationen, Vol. 35, page 261. These studies are of such practical interest that it is my intention to continue them during the coming year on an extended series of feeding experiments, in which I hope to interest experimentors in different parts of the country.

Mr. Lupton said that an investigation was now in progress in the Alabama State laboratory as to the effect produced by feeding cotton-seed meal. The results were not yet sufficiently large to justify the making of a formal report, but he had with him some results that it might be well to mention. So far he had found no diminution in volatile acids. For instance, he had 31.59, 31.79, 31.39 and 29.51, 29.44. 29.87; but the melting point was decidedly higher—37.5, 37.3, 37.6, 37.5, 37.6, 37.2. They were all registered Jersey cattle. He had not yet made an analysis of the butter, discarding cotton-seed meal and feeding on the ordinary feed. He made this presentation merely as confirming Messrs. Harrington's and Wiley's results as far as the increased melting point was concerned; he had not found it so in regard to volatile acid.

Mr. Wiley said that Jersey cows might furnish a butter even higher in volatile acids than that stated; therefore it could not be said that there was no diminution in the volatile acid unless they had been examined after feeding upon ordinary food. The results stated might be higher than ordinary.

Mr. Lupton said that the investigation would be continued.

The president said that the term "ordinary feed" was very pliable. What was ordinary feed in Maryland was not ordinary feed in Alabama, Texas, or Mississippi, where the variety of grass was very different from that found here, and the Lespedeza so largely used as pasture grass, especially in summer and sometimes preserved as hay, was a wonderful milk-producing grass. It was his impression that when the subject should be further investigated it would be found that the pasture grasses of the South affected very materially the condition of the butter product as compared with that of the North. The chemists in different parts of the country should combine for the solution of this troublesome problem. The combination between Messrs. Harrington and Wiley and the Maryland Station was a most excellent thing, and if Alabama would work with them or pursue the investigation on an independent line it would tend to throw light upon the matter.

Mr. Lupton said that the ration was carefully fixed and determined and that when he said ordinary feed he meant the ration fed to the cattle which was accurately known and would be reported in the final result.

Mr. Wiley said that by "ordinary feed" he meant to exclude cotton-seed meal. The only way to determine the influence of any feed was to exclude all other feed and that could be done for a few days at a time without affecting the health of the animal, but long enough to enable her to get the effect of the sample. It was self evident that these experiments must be confirmed upon the same animal and not upon a mixture, or if upon a mixture upon the same mixture.

Mr. Harrington said that there were two points as to which he was well convinced; one was the high melting point mentioned in the paper and the other was that the volatile acids were low. As to the former point he had no doubt at all, but the other made so radical a change in what had been supposed to be the normal amount of volatile acids in normal butter that he had secured the aid of Mr. Wiley in comparative work. He had then begun some experiments to determine the relative value of cotton-seed meal and boiled cotton-seed. He wanted to determine incidentally the value of these three foods as regarded milk production and at the same time the butter work could be carried along. When spring came he had found difficulty in keeping the animal on this exclusive feed; cotton-seed and cotton seed meal were extremely rich feeds, and in warm weather one cow sickened and one cow refused to eat, so that the results last spring were altogether thwarted. He had done a great deal of analytical work, but was not prepared to furnish the figures, as he proposed to repeat the entire experiment as soon as the weather was cold, when he should endeavor to put cows upon these various feeds, first analyzing the butter on ordinary feed, by which he meant other than cotton-seed or cotton-seed meal, and then feeding them on the cotton-seed for at least eight days.

Mr. Wiley presented that portion of the report on dairy products which related to the analysis of milk as follows:

ABSTRACTS OF DATA RELATING TO THE COMPOSITION AND ANALYSIS OF MILK, COLLATED SINCE THE LAST MEETING OF THE ASSOCIATION.

(1) Composition and Properties.

COMPOSITION OF CONDENSED MILK.

By J. C. Shenstone.

[Journal de Pharmacie et de Chimie, No. 6, September 15, 1889, p. 257.]

In the estimation of nitrogenous matters, the method of Ritthausen was employed. The fatty matters were separated by means of ether. The estimation of the sugars was made in the following manner: 30 grams of the condensed milk were dissolved in hot distilled water and allowed to cool. The solution was diluted to 97 cubic centimeters, and 3 cubic centimeters of a solution of nitrate of mercury added. When the separation of the caseine was completed the liquid was filtered. Ten cubic centimeters of the filtered liquid were taken and diluted to 100 cubic centimeters with distilled water, and the sugar therein estimated by Fehling solution. A portion of the same liquid was examined in the polariscope. The deviation observed is the sum of the deviations caused by the milk sugar and cane sugar present. The following table shows the limitations in the composition of ordinary condensed milks:

	Per cent.
Water	24.8 to 30.3
Fats	4.0 to 11.5
Albuminoids	10.67 to 12.6
Ash	1.90 to 2.42
Milk sugar	14.20 to 15.75
Cane sugar	29.95 to 39.90

COMPOSITION OF WINTER MILK.

[Bulletin No. 9, of the laboratory of the Inland-Revenue Department of Ottawa, Canada].

Thomas McFarlane, chief analyst, in this bulletin gives a record of the analyses of 267 samples of winter milk. Of these 71 samples were passed by the inspectors as genuine, and 196 were sent to Ottawa for analysis. Of the latter 87, or 32.5 per cent. of the number collected were found to be defective.

In regard to the unadulterated milks the following mean percentages of composition were found:

Total solids	12.58
Fat	3.79

No less than 55.6 per cent. of the number of milks which they condemned as doubtful by the lactoscope proved to be so on chemical examination.

Dr. McFarlane says, in closing, these figures show that in the hands of an unpracticed person the lactoscope can not be depended on for always indicating closely the percentage of butter fat in milk, or furnish adequate proof of the degree of adulteration.

RELATIVE AMOUNT OF FAT IN FIRST AND LAST MILKINGS.

In regard to the difference in the percentage of fat in a milking, when the milk was divided as evenly as possible into two portions, it was found that the second portion contained 48.12 per cent. more fat than the first of the morning milking. For the evening milking the second milking contained 37.11 per cent. more. The mean increase for both morning and evening milkings was 43.55 per cent. The average composition of the milk standing over a period of ten weeks is represented by the following figures:

	Water.	Fat.	Ash.
First period:			
Morning milking	86.81	3.90	.77
Evening milking	87.24	3.86	.74
Second period:			
Morning milking	86.63	3.73	.73
Evening milking	86.86	3.99	.74
Third period:			
Morning milking	86.71	4.11	.75
Evening milking	86.90	4.01	.74

NOTE.—The solids not fat in the above analyses are as follows:

No. 1 .... 13.19 — 3.90 = 9.29
No. 2 .... 12.76 — 3.86 = 8.90
No. 3 .... 13.37 — 3.73 = 9.64
No. 4 .... 13.14 — 3.99 = 9.15
No. 5 .... 13.29 — 4.11 = 9.18
No. 6 .... 13.10 — 4.01 = 9.09

From the above numbers it will be seen that the variation in solids not fat are much larger in the samples examined above than are usually found. (Wulff, Bulletin No. 24, Experiment Station of Indiana.)

BEHAVIOR OF THE SALTS OF MILK WITH CASEINE.

An analysis of the ash of milk gives the following mean numbers in grams per liter of milk:

	Grams.
Chlorine	0.90
Phosphoric acid	2.42
Potash	1.80
Soda	0.49
Lime	1.90
Magnesia	0.20

Sulphuric acid does not exist in the original salts of the milk, but is produced from the sulphur of the albuminoids. Caseine seems to form two distinct compounds with lime; one contains 2.39 per cent. of lime, showing an alkaline reaction with litmus, while the other compound only contains 1.55 per cent. of lime. The amount of acid reaction in caseine was determined by titration with soda, using phenolphthalein as indicator. From the various reactions studied it appears that the caseine is naturally present in milk as a calcium salt with 1.55 per cent. of lime. The excess of base in the milk is probably present as organic salts, citric acid being a constant constituent of milk to the extent of 1 gram per liter. The acidity of milk to phenolphthalein is probably due to the presence of acid phosphates. Calculated from the probable combination of the bases in the original milk the salts present in milk may be grouped as follows, the numbers expressing grams per liter:

	Grams.
$NaCl$	.962
KCl	.830
KH_2PO_4	1.156
$K_2H PO_4$	.853
Potassium citrate	.495
Dimagnesium phosphate	.336
Magnesium citrate	.367
Dicalcium phosphate	.671
Tricalcium phosphate	.806
Calcium citrate	2.133
Lime (in caseine)	.465

Hammarsten considers that caseine acts as a solvent for calcium phosphate while Eugling believes that the caseine enters into combination with tricalcium phosphate, The latter theory is rejected as being based on erroneous suppositions. Determinations of lime and phosphoric acid were made in the serum of milk filtered through porous cells and in the insoluble portion of milk. From 36 to 56 per cent. of the phosphoric acid and from 53 to 72 per cent. of the lime were found to be insoluble. It was found that 85 per cent. of calcium in milk is precipitated by ammonium oxalate. The reason that boiled milk will either not curdle at all or requires a longer time to curdle than fresh milk is that a part of the dissolved calcium salt is precipitated as tricalcium phosphate. For the same reason the curdling of milk by rennet is prevented or retarded by adding more or less alkali. In either case the property of being curdled by rennet may be restored to the milk by adding acid, passing carbonic anhydride through it or by the addition of soluble calcium salt. (Soldner Landw. Versuchs-Stat., 35, 351–436.)

ASSIMILATION OF MILK SUGAR.

[Bernard: Lecons sur les phenomènes de la Vie, vol. 11, page 543.]

Milk sugar, according to Dastre, is not directly assimilable. When injected into the veins of a dog it is recovered entire in the urine.

To be used as bodily food lactose must undergo a transformation, *i. e.*, be changed into dextrose and galactose.

A man suffering from diabetes was fed on a diet from which every sugar except lactose was carefully excluded. The sugar recovered in the urine was wholly dextrine—having in a pure state a specific rotary power. AD = 52°.4.

As the dose of lactose was increased the quantity of dextrose in the urine was larger; whence it follows that lactose, when entering the system of a glycosure through the digestive organs, appears only as dextrose in the urine. The quantity of dextrose, however, is not equal to the whole of the lactose exhibited. In the case cited, the quantity of dextrose in the urine per twenty-four hours, when no lactose was used except what was in the milk, was 5.72 grams; when 50 grams lactose were added, 62.30 grams; 100 grams lactose, 83.60 grams; 200 grams laetose, 127.95 grams.

The questions which arise are the following: (1) Is this transformation of the glucose a process which takes place directly in the alimentary canal? (2) Or have the products of digestion served to form temporarily some glycogene which is subsequently decomposed, forming dextrose?

While the present state of our knowledge will not permit us to answer these questions, the facts cited lend additional probability to the hypothesis that dextrose (glucose) is the final state to which all sugars are reduced in digestion.

MILK SUPPLY OF PARIS.

[Pabst: Revue International des Falsifications, 1889, Vol. 2, page 139.]

In 1881, 50 per cent. of the milk delivered in Paris was skimmed or watered. In 1882 this proportion was reduced to 30 per cent., and in 1887 to 16.6 per cent. This improvement was due to the supervision of the municipal laboratory. The mean percentage of solids in the milk of Paris is 13, of which 4 per cent. is fat and 5 per cent. milk sugar. The lowest permissible percentage of solids is 11.5, with 2.7 per cent. fat. According to the rules established by the laboratory, such milk would correspond to an addition of 11.6 per cent. of water. It is concluded from a study of the analytical data that better milk can be had in Paris than in any other European city.

SOLIDS IN MILK.

[Faber: Chem. News, May 24, 1889, page 253.]

The mean of 50,000 analyses of the Dairy Supply Company of Denmark shows solids, not fat, of 87 to 88 per cent.

The variations in total solids is due almost wholly to variations in fat. Evening milk contains more fat than morning milk. In October and November the milk is richer in fat than at any other season.

COMPOSITION OF BUFFALO MILK.

[Szentkiralyi: Oestereicher Landwirtschafliche Wochenblatt, 1889, No. 11, page 83. Bied.: Central Blatt, May, 1889, page 348.]

The mean composition of buffalo milk compiled from a large number of analyses is as follows:

	Per cent.
Dry matter	17.85
Fat	7.87
Protein	5.05
Lactose	4.52
Ash	0.76

NOTE.—In the above analyses it will be seen that the sum of the several mean constituents shows 18.18 per cent. of dry matter, while the mean given in the table is only

17.81 per cent. In many of the analyses given, however, the dry matter has not been determined, notably in one case, where the fat was 9.19 per cent. In spite of the large content of fat the specific gravity of buffalo milk is somewhat higher than cow milk. The maximum number given is 1.0349, and the minimum, 1.0310.—H.W.W.

COMPOSITION OF MILK ON ONTARIO EXPERIMENT FARM.

[James: Bulletin, Ontario Experiment Station, No. 39.]

Ninety-two samples were analyzed with the following mean results:

	Per cent.
Water	87. 18
Albuminoids and sugar	8. 08
Fat	4. 03
Ash	. 70
Total dry matter	12. 81

MEAN COMPOSITION OF MILK.

[Fleischman: Milk Zeitung, Vol. 15, page 5.]

Period.	Specific gravity.		Dry matter.		Fat.	
	Morning.	Night.	Morning.	Night.	Morning.	Night.
January 1 to May 19	1. 0313	1. 0313	11. 96	11. 97	3. 13	3. 15
May 19 to October 18	1. 0307	1. 0306	12. 10	12. 21	3. 35	3. 41
October 18 to December 31	1. 0314	1. 0313	12. 09	12. 14	3. 38	3. 40
Mean	1. 0311	1. 0310	12. 05	12. 11	3. 29	3. 32

For the whole day the means are as follows:

Specific gravity at 15°		1. 03104
Dry matter	per cent	12. 077
Fat	do	3. 304
Daily product per cow	kilograms	7

REPORT ON MILK FROM CANSTADT.

By Dr. J. N. ZEITLER.

[Zeitschrift für angewandte Chemie, 1889, page 13.]

Fifty-seven analyses of milk were made ranging from the 9th of March until the 11th of August. The samples were partly morning and partly evening milk. The analytical data were confined to the determination of the specific gravity of the whole milk, and the skimmed milk at 15°. The estimation of the fat by extraction of ether and weighing and by the areometric system of Soxhlet, determination of the total solid matter in the milk in about one-half the samples.

The variations observed in the samples of milk were as follows:

	Specific gravity of the full milk.	Specific gravity of the skimmed milk.
Least	1. 0275	1. 0293
Highest	1. 034	1. 036

In general the fat estimations determined by the two methods were nearly identical. The least fat determination was 2.42 per cent., and the highest 4.97.

Total solids varied as follows: The least percentage of total solids was 10.52 and the highest 13.97.

THE LIMITS OF SPECIFIC GRAVITY OF COW'S MILK.

By M. Schrodt.

[Chem. Cent.-Blatt, 15 December, 1888, page 1560.]

In morning's milk the range of specific gravity was from 1.0290 to 1.0342. For evening milk the range was found to vary from 1.0284 to 1.0349. A density of 1.033 was found in the morning milk in six cases and in evening milk in twenty-two cases, the milk having been drawn from ten to fifteen cows. Evening milk with a density of 1.034 and above was found eight times, with milk from five to nine cows. The mean density for the whole year for all the samples of milk was 1.03162.

FAT IN MILK.

[Abstract in Chem. Zeit. No. 52, 1889, page 853.]

Bechamp (Bull. Soc. Chem., Paris, May 24, 1889) contends that the fat particles do not float freely in milk, but that each particle is inclosed in a cell of an albuminoid nature.

When milk is mixed carefully with ether so as to produce no emulsion or foam, and set aside, the liquids are separated. It is found that the fat particles in the lower layer have increased greatly in size and their appearance has been changed. The volume of the ether layer also diminishes, whereas should the fat dissolve in the ether the volume of the latter would be increased. The volume of the milk is found to be correspondingly increased. It also parts into two layers, an upper one containing the particles having absorbed ether, and a lower liquid layer.

NATURE OF MILK.

By A. Bechamp.

[Comptes Rendus, Volume 107, page 772.]

This paper contains a description of the properties of milk in which it is regarded as the product of a physiological function which takes place as the sequence of other functions in glands. It is maintained that the milk is not an emulsion, but that the fat particles, as Dumas supposed, are surrounded by a membrane which hinders their solution in ether. It is shown in caseine as a definite chemical compound and that the milk contains other well characterized albuminoids. Milk coagulates of its own accord without the addition of any foreign ferments. The liquid portion of the milk contains no free caseine in solution or suspension, but the caseine is contained with other albuminoids and with alkalis. In other words, the milk contains caseine in the same way that a solution of sulphate of potash contains sulphuric acid. The alkaline caseinates and the albuminates of the milk are not precipitated by alcohol of medium concentration. The membrane which surrounds the fat particles is not soluble in a diluted solution of sesqui-carbonate of ammonia, but caseine is soluble in this solution. For the extraction of the fat particles, the milk is poured into a sufficient quantity of dilute alcohol, filtered, and the residue washed with alcohol and sesqui-carbonate of ammonia in order to remove caseine and other albuminoids. The isolated mass of these particles does not resemble butter. They remain intact in a solution of sesqui-carbonate of ammonia or 30 per cent. alcohol. The dried mass does not completely melt, the unmelted portion retaining butter like a scum. After treatment with ether, there is left 1.3 per cent. of the weight of the dried butter fat cells, and this is not caseine, but probably of an epidermic nature. The incinerated fat particles leave behind an ash. The dried particles are destroyed by a treatment with ether, the butter being dissolved.

FIBRINE IN MILK.

[S. M. Babcock, Proceedings of the Society for the Promotion of Agricultural Science, 1888, page 13.]

Fibrine was detected in milk by the reactions which it gave with peroxide of hydrogen and with gum guiacum. The peroxide of hydrogen test was applied as follows: A solution of peroxide of hydrogen was added to the milk and the mixture tested from time to time. After a certain period it was found that the peroxide of hydrogen had entirely disappeared, this disappearance being accompanied by a slight effervescence showing the escape of oxygen. Some milks were found to be more active than others, but all responded to this test. Cream is more active than milk and skimmed milk gives almost no reaction, showing that the principal amount of the fibrine is removed with the cream.

The gum guiacum test was not directly applied to milk, as the blue color is masked by the milk, but if a piece of filter paper is moistened with milk and dried and the test applied to the paper it will be colored faintly blue in spots after a short time. Milk which has been heated to near the boiling point for a short time loses its power of decomposing peroxide of hydrogen. The effect of fibrine in the cream is to render churning difficult and the recovery of fat imperfect, and there is little doubt that the beneficial effect of ripening of cream is due to the decomposition of the fibrine which takes place during this process. Cream that has not been ripened may be churned without loss by adding to it a suitable quantity of lactic or acetic acid, and it is believed that muriatic or phosphoric acid will accomplish the same end. There is a tendency in imperfectly ripened cream for the fibrine to adhere to the butter. If butter made in this way be melted and filtered and residue washed with ether it will be found to give the reaction for fibrine. The quantity of fibrine present in milk is usually very small. It is probably not more than .002 to .003 of 1 per cent.

In order to discriminate between the fibrine of milk and that of blood it is proposed to call this milk fibrine lactofibrine.

CITRIC ACID IN MILK.

By F. Sonlhet.

[Wiener landwirthschaftliche Zeitung, 1888, page 401; and Journal de Pharmacie et de Chimie, 1st November, 1889, page 405.]

The investigations of Mr. Sonlhet were carried on under his direction by Mr. T. Haeckel.

Citric acid is found as a normal constituent of cow's milk. The analyses of a great number of samples of widely different sources disclosed the presence of from 1.8 to 2.2 grams of citrate of lime in a litre of milk, corresponding to .9 to 1.1 grams of citric acid. This equivalent to a mean content of .1 per cent. of citric acid in the milk. The milk from one good cow furnishes each day as much citric acid as there is in two or three lemons.

The concretions which are frequently met with in, concentrated milk are almost pure citrate of lime.

Woman's milk does not contain citric acid, and its occurrence is probably peculiar to herbivores.

The discovery of citric acid in the milk fills a gap which has long been regretted in the composition of this substance. It is known, indeed, that the serum of milk holds in solution a greater quantity of lime than was necessary to establish a proportion between the mineral acids and the bases; and in the discovery of citric acid this fact finds now its first natural explanation.

The citric acid of the milk comes, without doubt, from the vegetable food of the cow, either the green and dry fodder containing a greater or less quantity of it, or from the fact that it appears at the same time as converted vegetable acids during the fermentation of the cellulose.

CHEESE AND ICE-CREAM.

[De Vries, Milch Zeit. October, 1888, pages 861–862 and 881 to 888.]

Blue cheese.—The blue color is caused by bacteria. The blue color is removed by the oxygen of the air. These bacteria are different from those in blue milk.

Tyrotoxikon in Ice Cream.—Vaughn[1] has described symptoms of poisoning of persons who had eaten vanilla ice cream. The vanilla was shown to be absolutely free from any toxic quality, the milk, however, from which the cream had been made had been kept in a place where the formation of tyrotoxikon was possible. This ptömaine is determined as follows:

The milk is coagulated and filtered; the filtered liquid is rendered slightly alkaline by potash lye and agitated with an equal volume of anhydrous ether. After some hours the ether is removed with the aid of a pipette and left to spontaneous evaporation. The tyrotoxikon remains in the form of needle-like crystals.[1]

(2) Apparatus.

THE LACTOMETER; ITS UTILITY AND RELIABILITY.

Under this title, Dr. Charles E. Munsell, has published a paper on the use of the lactometer which is published in the Journal of the American Chemical Society, Vol. 10, No. 6.

The following is an abstract of the most important points discussed by Dr. Munsell. He says that in order to obtain scientific accuracy with the lactometer, it is necessary to have an instrument which is absolutely correct, that is, exact from 90 to 110 degrees and not over one degree in error at other points; and the examiner must be an expert, who uses all the precautions that would be needed in employing other sensitive instruments, for the lactometer is the most delicate hydrometer in ordinary use, each degree being less than one-twentieth of a degree Baumè, its modulud being the highest, 3,548$\frac{8}{29}$, while that of the Baumé scale is 149.

Lactometers as obtained from the maker, should be tested at 60° Fahr.; this is done either by comparing with a standard lactometer, which is known to be absolutely correct (this is the best method when only one instrument is to be examined), or by means of standard solutions; these solutions are made of salt or dilute sulphuric acid. A 3 per cent. solution of sodium chloride has a specific gravity of 1.0217 (75°); a 4 per cent. 1.029 (100°); and a 5 per cent. 1.036 (122°); these solutions, and others at 90° and 110° should be tested with a specific gravity bottle at 60° Fahr.

With sulphuric acid a 3 per cent. solution has a specific gravity of 1.019 (66°); a 4 per cent. 1.0256 (88°); a 5 per cent. 1.032 (111°).

The fault with the salt solutions is that they vary from evaporation; the dilute sulphuric acid solutions are more constant, still they should be tested occasionally.

It is necessary to allow the lactometer to descend into the milk slowly, as the milk which remains on the portion of the stem above the surface of the sample being tested, when the entire stem is dipped, would lower the reading about two degrees; the sample should not be shaken more than is necessary to mix any cream which may have risen, as it will absorb air; and the foam on the surface should be removed, as it makes it difficult to read the scale.

For accurate tests the sample should be cooled (if above 60° Fahr.) by placing the bottle or cylinder in cold water, or packing with ice; or if below 60° Fahr. by setting in warm water, rubbing the cylinder with the hand, or placing in a warm room. A difference in temperature of from 2½ to 3½ degrees Fahrenheit will cause a variation of one lactometric degree, different samples of milk having different co-efficients of expansion; 3 degrees is the usual average, and is approximately correct from 40 to 70 degs. Fahr.

---

[1] Journal de Pharmacie et de Chimie, 1889, page 164. Ueber die Anwesenheit von Tyrotoxikon im giftigen Eis, etc., Arch. f. Hyg., from Arch. der Pharm., (3) XXVI, 1888, page 1089.

Beyond these temperatures its calculation is uncertain, particularly if the temperature is near freezing, or so high that the milk is liable to sour; when once frozen the constituents of the milk appear to separate, and after thawing the specific gravity is different.

When the contents of a can of milk are frozen, the water freezes first, pushing the cream and caseine towards the center, in very cold weather forming an inner core of cream; while, after milking, the ice around the exterior has a specific gravity sometimes as low as 1.008; a common trick of milk dealers being to pour out the core and then scrape out the ice, some times mixing it with snow or ice.

The following formula for a compound for circumventing the lactometer was devised by the chemist who was employed by the Hester street coalition of dishonest dealers:

(1) One gallon of water,
(3) Three ounces of sugar,
(1½) One and a half ounces of salt, and a little caramel;

This to be added to

(4) Four gallons of milk.

This gives a fluid which stands at 99 degrees on the lactometer at 60 degrees Fahrenheit, and shows on analysis:

	Per cent.
Water	89.06
Fat	2.95
Caseine and sugar	6.80
Salt	1.19
Sodium chloride	0.42

The salt can be easily detected by its brackish taste, and the dealer who offered it for sale did the State one month's service besides paying a fine.

Dr. Munsell closes his paper with an elaborate discussion of the formula for finding the error of a lactometric degree from the true position of the same, according to the laws of graduation of hydrometers by the specific gravity. This discussion is followed by an elaborate table, giving the error of the lactometer for each degree in centimeters, the distance from zero to 100 being one meter. These tables are found on pages 131 and following, of the Journal of the American Chemical Society, already cited.

NEW BUTYROMETER.

[Gerber. Milch. Zeit., Vol. 18, page 265; Chem. Central Blatt, 1889, Vol. 20, page 657.]

The instrument consists of a tube 25 centimeters in length, and 15 centimeters in diameter in the graduated portion. The tube above is expanded to twice the given diameter and the neck is constructed to receive a cork stopper. The graduation is in one-tenth cubic centimeters and extends to the expanded portion. The scale is so arranged that milk both rich and poor in fat can be treated. The ether 10 cubic centimeters, alcohol 10 cubic centimeters, and alkali solution a few drops, are first added, and then the 10 cubic centimeters of milk to be tested. A single shaking is all that is necessary. The curd separates in very fine particles, which is a matter of considerable importance. The instrument is placed inverted on the stopper in the water-bath. It is made by Kramer in Zurich.

(3) Analytical Methods.

ESTIMATION OF THE CONTENT OF FAT IN MILK BY THE METHOD OF SOXHLET-ENGSTRÖM AND BY THE METHOD OF SCHMOEGER-NEUBERT.

By M. Kühn.

[Milch Zeitung, July 31, 1889, page 601.]

As is well known in the ordinary areometric method of Soxhlet milk which has been transported for a long distance or which has stood upon ice, also freshly separated centrifugal milk and sweet buttermilk afford a very imperfect separation of the ether fat solution. Dr. Soxhlet therefore has proposed the well-known modification of the method in which he adds to the sample of milk to be determined 10 grams of sulphate of potash, or the separation of the ether fat solution in warm water, or the use of 20 cubic centimeters of potash ley, etc. Dr. Engström has also proposed the following modification of the process: To 200 cubic centimeters of milk add 20 to 30 drops of glacial acetic acid and shake vigorously. Add then 60 cubic centimeters of ether and shake for half a minute. Afterwards are added 13 to 15 cubic centimeters of the potash solution, and the sample is then treated by the usual method for the separation in Soxhlet's apparatus. Kühn's modification is as follows: The determination of fat in the fresh, whole, or skimmed milk is made by evaporating 10 cubic centimeters of the milk with about 30 grams of sea sand or burned gypsum and extraction with ether for three or four hours. The milk which has stood in glass flasks for about twenty-four hours on ice is warmed to 17.5° and carefully skimmed. The fat was then determined in it according to the original method of Soxhlet. With skimmed milk this attempt usually failed, and this often also happened for full milk on account of the difficulty of separating the ether fat solution. The fat was determined in the same samples by the Engström method. In most cases the separation of the ether took place in sufficient quantity in from three-quarters of an hour to an hour. A table is given of comparative analyses in which it is shown that on whole milk Soxhlet's original method agrees very well with the gravimetric method, while the process of Engström shows a considerably lower result. With skimmed milk, however, the process of Engström agrees very well with the gravimetrie methods. Determinations were also made by the variation of the method proposed by Drs. Schmoeger and Neubert, the so-called Proskauer method, which consists in the solution of 10 grams of sulphate of potash in the milk. By this process the separation of the ether fat solution takes place much quicker and better, and the milk after the addition of the sulphate of potash should be strongly shaken until the butter begins to separate. The method, however, seems to be much better adapted to skimmed milk than to whole milk.

DETERMINATION OF THE FAT IN MILK BY THE WERNER-SCHMID METHOD.

By A. W. Stokes.

[Analyst, February, 1889, page 29.]

Mr. Stokes reports that he has tried various methods of estimating fat in milk, the method of Wanklyn, the lactobutryuometer, Adams method, and the lactotrite.

None of these methods has been found perfectly satisfactory. The two former are not accurate; the paper coil requires too much time, and the lactotrite is too expensive and unsuitable for a laboratory. He has therefore tried the Werner-Schmid method,[1] which is as follows:

"Dr. Werner-Schmid takes a test-tube of about 50 cubic centimeter capacity, graduated in tenths of a cubic centimeter; introduces 5 cubic centimeters of cream or 10 cubic centimeters of milk, accurately measured; adds 10 cubic centimeters of strong hydro chloric acid; boils, with shaking, until the liquid turns dark brown; cools by placing the tube in cold water, adds 30 cubic centimeters of ether, shakes round, lets stand, measures the volume of ethereal solution, draws off 10 cubic centimeters with a pipette, evaporates down in a weighed porcelain capsule on the water-bath, and finally in an air-bath at 100°. He then weighs and calculates for the original quantity of the ethereal solution. If the process has been rightly conducted the ether separates from the aqueous solution clear, without the slightest turpidity. The ethereal solution, as it flows out of the pipette, should not show any watery drops. The results are perfectly accurate, and differ from each other and from the ordinary gravimetric methods by less than one-tenth per cent. The operation requires at most fifteen minutes."

---

[1] Bull. 19, Chem. Div., p. 8.

Some ordinary samples of milk gave the following percentages of fat by the various methods employed. These percentages are compared with the calculation of the fat from the specific gravity and total solid estimations:

	Wanklyn.	Adams.	Schmid.	Calculation.
No. 1	2.64	2.91	2.90	3.14
No. 2	2.53	2.71	2.70	3.11
No. 3	....	2.48	2.37	2.95
No. 4	2.04	....	2.53	2.70
No. 5	2.71	....	3.08	....
No. 6	....	....	2.88	3.22
No. 7	....	....	3.48	3.61

Skimmed milks were also tried, and with good results. The following results were obtained with creams:

	Wanklyn.	Adams.	Schmid.
No. 18	42.8	....	42.4
No. 19	....	52.8	52.5

The action of Werner-Schmid process is found in the total destruction of the caseine by the acid, while the fat is set free and rises to the surface. The ether dissolves this fat, but perhaps not entirely. There is always a minute amount of fat present in the hydro chloric solution. The method seems to possess all the advantages, speed, simplicity, accuracy, cheapness of apparatus, and little loss of re-agents. Calibrated tubes made for the purpose may be obtained of Messrs. Cetti, Holborn, London.

PROCESS FOR ESTIMATING FAT IN MILK BY THE GRAVIMETRIC METHOD.

By Th. Dietrich.

[Zeitschrift für angewandte Chemie, 1889, part 15, page 413.]

A strip of filter paper 27 centimeters in length and 8 centimeters in breadth is wound around a cylinder 28 millimeters in diameter, by means of which a cylindrical shell is made of the paper 5 or 6 centimeters high, the under part being closed by pressing the paper together. In like manner a similar cylinder is made of wadding which fits exactly into the filter paper cylinder described. Pure white wadding is employed, obtained from M. Küstermann's successor in Freiburg. This wadding is cut into appropriate lengths and wound around a cylinder of 20 millimeters in diameter. By a few blows from the wooden cylinder on which the wadding has been wound, the part at the bottom is pressed together into a tight mass. We have formed thus a double shell, the outside of filter paper and the inside of wadding. The thickness of the wadding amounts to 4 or 5 millimeters. The interior of the cylinder can be half filled with loose wadding. If the milk to be used is sour it should be shaken with a few drops of ammonia until a homogeneous liquid is secured. From a weighing bottle some 15 to 20 grams of the milk are poured upon the prepared cylinder. The cylinders are then dried in small glass dishes with flat bottoms, at 60° to 80°. The glass dishes have a double purpose, first, as a holder for the cylinders; and second, to receive any of the milk which might percolate through. The latter accident only happens when the cylinders have not been carefully made. The extraction of the fat is subsequently carried on in the usual way. The paper and wadding used should be carefully extracted beforehand or the amount of extract in them determined and deducted from the total weight of fat obtained. An extraction apparatus is used which is connected above and below by ground glass connections, the apparatus being furnished by E. Greiner, of Stutzenbach.

RÖSE'S METHOD OF ESTIMATING FAT IN MILK.

[Bericht über die Thätigkeit des Milchwirthschaftlichen Instituts zu Proskau, 1888–'89, pages 11, 12, 13, and 14.]

About 20 cubic centimeters milk are exactly weighed in a burette, with a few cubic centimeters water, 2 cubic centimeters ammonia, 45 cubic centimeters alcohol, and 60 to 65 cubic centimeters of ether and petroleum spirit and shaken together. After standing for some time two sharply defined layers are formed, the under one consisting of serum, and the upper one, which is composed of a mixture of alcohol ether and petroleum spirit, contains all the fat in the milk except a mere trace remaining in the serum. The volume of the upper layer is exactly read off on the burette, and the aliquot part of it removed by means of a pipette, and the fat contained therein estimated after the removal of the volatile substances. By calculation from the total amount of material in the upper layer, the quantity of fat contained in the milk is determined.

This method of the estimation of the fat was compared with the Adams method, the paper coils having been extracted by means of ether for one hour. The following comparative results were obtained:

Example.	Adams's method.	Röse's method.
	Per cent.	*Per cent.*
1	2.45	2.34
2*	.30	.29
3	3.09	3.04
4	5.19	5.17
5	4.99	5.01
6	1.77	1.79

*Skimmed milk.

The method of Röse was also extended so as to include curdled milk, and the results were found to be equally as accurate as in the foregoing cases. It was also tried with milk to which borax had been added and allowed to stand for three-quarters of a day. The results were also good. It was tried further with milk to which borax was added and allowed to stand until sour, and the results were still satisfactory.

In the same pamphlet, page 15, is found the result of a study of the estimation of fat by previous drying of gypsum, and subjected to different periods on extraction. It is shown that the period of extraction has a great deal to do with the accuracy of the method, the increase in the amount of fat obtained, however, after three hours being very slow. As an illustration of this, one experiment may be cited:

Amount of fat extracted by previous drying on gypsum.

	Per cent.
Extracted for from one-half hour to one hour.....	.99
Extracted for three hours..........................	2.63
Extracted for five hours..........................	2.71
Extracted for ten hours..........................	2.76
By the Adams method..........................	2.81

Further experiments are described on pages 16 and 17 in the use of ignited strips of asbestus instead of paper in the estimation of fat in milk by the Adams method. The results obtained show that with the asbestus slips almost the maximum extraction of fat was obtained after one hour; for instance, in one experiment the percentage obtained after one hour was 3.08, and after three hours, 3.13. The method also agreed closely with the Adams method; for instance, by the asbestus method 3.17, by the Adams method 3.14. It was even found that one-half hour extraction was usually sufficient to remove the whole of the fat, as is illustrated in the following figures:

	Per cent.
Extraction for half an hour..........................	3.09
Extraction for four hours..........................	3.14

With skimmed milk the results were equally as good; for instance:

	Per cent.
Extraction for one-half hour..........................	.16
Extraction for four hours..........................	.20

A series of fifteen experiments recorded on page 19 fully corroborates the numbers given above. A comparison of the results obtained by the Adams method and the old gypsum and Soxhlet methods is given on pages 22 and 23. Some of the results obtained are as follows:

Adams method.	Gypsum method.	Soxhlet method.
2.87..................	2.79	2.73
4.16..................	3.89	3.93
2.89..................	2.63	2.74
4.15..................	3.90	3.87

As the result of a very extensive series of analyses, for the details of which I refer to the pamphlet described, the following conclusion was drawn: "Therefore, it is believed that all results obtained by methods previous to the introduction of the Adams method for the estimation of the amount of fat in full milk give that amount in a per cent. too low by from .1 to .15."

ESTIMATION OF THE CONTENT OF FAT IN CURDLED MILK.

By M. Kuehn.

[Chemiker-Zeitung (Chemisches Repertorium), 1889, No. 66, page 228.]

The curdled milk is rendered fluid with 40 per cent. potash ley, the re-action being made slightly alkaline. For the absorption of the milk preparatory to drying, the following mixture is used: 25 grams of burnt gypsum, 4 grams precipitated carbonate of lime, and 2 grams acid sulphate of potassium. These three salts are previously intimately mixed. The acid sulphate of potassium protects the milk fat from saponification by the excess of potash ley, and the carbonate of potash is for the purpose of converting the excess of sulphuric acid into acid potassium sulphate. Ten grams of the milk are quickly weighed in a covered measuring glass, the milk is poured over the mixed powder, and the glass again weighed. The mixture is now dried on a water-bath and the lumps therein contained rubbed up as finely as possible with a glass rod. The dried mass is finally reduced to a fine powder and placed in a paper cylinder, previously extracted, and the fat removed by treatment in a continuous extractor for four hours with ether. If the obtained extract is not entirely clear it must be filtered. The ether is then distilled off and the fat dried from one to two hours in a drying chamber. The quantity of gypsum removed in the extract, amounting to from 2 to 4 milligrams, is neglected in the calculations. (Abstracted from the Milch Zeitung, 1889, Vol. 18, page 561.)

ESTIMATION OF FAT IN MILK.

By H. Droop Richmond.

[The Analyst, July, 1889, page 121.]

The estimation of fat in milk, simple though it seems at first sight, has perhaps given more trouble to analysts than anything else. It has been pointed out by Vieth that the difference between the percentages of fat extracted if the Adams process is used, and the fat extracted if the plaster process is used, is smallest with skimmed milks, and increases with the increasing percentage of fat. The author therefore undertook a comparison of the different methods of fat extraction, which he classifies under the following fifteen heads:

(1) Wanklyn's direct extraction, and modifications by Carter Bell, Waller, etc.
(2) Evaporation on sea sand.
(3) Evaporation on Asbestus. Babcock (Department Agriculture Report, 13, part I, 81.)
(4) Evaporation on asbestus cloth or paper. Wiley (Foods and Food Adulterations, Part I., page 81), also used by Johnstone (Analyst, XII, 234).
(5) Evaporation on plaster of Paris. Soxhlet.
(6) Evaporation on pumice, with grinding to powder. Storch (Analyst, XII, 6).
(7) Evaporation on copper sulphate. Morse, Piggott, and Burton (Analyst, XIII, 18).
(8) Evaporation on sponge. Duclaux (LeLait, page 176).
(9) Evaporation on lint. Abrahams (Analyst, IX, 20).
(10) Evaporation on paper. Adams (Analyst, X, 46).
(11) Evaporation on powdered glass. Baynes.
(12) Evaporation on kieselguhr.
(13) Evaporation on wood fiber. Gantter (Z. f. Anal. Chem.).
(14) Extraction from milk solids in pasty condition. J. Bell (Foods, Vol. II; and Analyst, VIII, 141).
(15) Extraction from precipitated casein.

Since it was evidently impracticable to compare all these methods, the following method of selection was employed:

(1) Wanklyn's method and modifications are now universally admitted to be faulty.

(2) By the use of sea sand, Schrott and Hengold find results very nearly the same as Soxhlet's plaster and areometric methods; other experimenters find rather less.

(3) Kent and Grothe (Analyst, x, 106) find that Babcock's method gives only slightly more than Wanklyn's, and .2 to .3 less than Soxhlet's.

(4) Wiley has discarded his asbestus paper method on account of it not giving results as high as Soxhlet's areometric method; Johnstone has published figures (Analyst, XII, 234) showing that results obtained by asbestos cloth fall appreciably below those given by Fleischmann's formula.

(5) Soxhlet's method, which is the usual method of fat estimation on the continent, gives very concordant results, which are higher than any of the preceding.

(6) Storch's method gives on an average .27 higher than Soxhlet's (Analyst, XIII, 6; Versuch. Stat., 1888, page 316).

(7, 8, 9, 11, 13, and 15) No comparative figures published for any of these methods.

(10) Adams's paper method gives higher results than any other, except Storch's (Analyst, x, 46; Baertling, Rep. f. Anal. Chem. 41 (1886), No. 31, 411, etc.).

(12) Neither method nor figures yet published.

(14) Bell's method has been examined by Wigner (Analyst, VIII, 211), and gives .2 to .3 more than Wanklyn's.

Richmond therefore decided to submit only three methods to comparative examination, namely: Soxhlet's plaster method, Adams's paper method, and Storch's pumice method, or as it is known, the Kieselguhr method. The results of the comparative analyses are found on page 124 of the number of the Analyst already cited. These results lead to the following conclusions:

(1) If a sufficiently long time is given for the extraction, and if the plaster and Kieselguhr are ground to a fine enough powder, there is, practically speaking, no difference between the paper, plaster, or Kieselguhr methods.

(2) The use of unextracted coils induces an error which is by no means constant, varying from +.05 to +.55. The observation that acids rendered a larger portion of the extractive matter of the coils soluble than would otherwise have dissolved led me to the conclusion that the free acid in milk (always present) increased the amount extracted from the coil itself, and this was further shown by the following experiment: A skim milk, when fresh, gave as a mean of three results .15 per cent. fat. A day or two afterwards, when beginning to turn sour, the fat had increased to .28 per cent., the correction of .010 grams being made for each coil.

THE ESTIMATION OF THE FAT IN MILK BY SOXHLET'S AREOMETRIC METHOD.

By J. KLEIN.

[Chemisches Repertorium, November 24, 1888, page 309.]

Klein replies to Soxhlet's objection to his former paper with the statement that the strips of paper used by him were previously exhausted of fat. He therefore insists upon his former statements in regard to the correction which should be applied to the Soxhlet tables which were published in the Chemisches Repertorium for 1888, No. 12, page 290.

DETERMINATION OF FAT IN MILK.

METHOD OF PATRICK.

Prof. Patrick, of the Iowa Experiment Station, has modified the method of separating fat from milk used in determinations by the lactocrict in the following manner:

The acid used for dissolving the caseine is a mixture of strong acetic, sulphuric, and hydrochloric acids in the respective proportions of 9, 5, and 2 by volume. The operation is conducted in a glass tube about three-fourths inches in diameter, 12 inches long, closed at one end by having a narrow construction about 3 inches long near the middle where the fat is to be read. 108 cubic centimeters of the milk are put into the tube and about 14 to 16 cubic centimeters of the mixed acid above mentioned added and the contents of the tube mixed by shaking. The tube is then placed upon a sand bath or some other convenient means of heating, and the mixture brought to the boiling point. Bumping is avoided by placing a bit of pumice stone in the tube. The boiling is continued for twenty minutes, by which time the milk will become brown and the fat will flow in a clear oily layer on the surface. The length of the column of fat is measured by a scale and its percentage calculated from a table compared with the gravimetric method and competent results have been obtained, as will be seen by the following table:

	By gravimetric method.	By new method.
	Per cent.	*Per cent.*
(*a*) On whole milk	3.33	3.35 3.42 3.33 3.35 3.40 3.31
	4.09	4.00 4.00
	3.72	3.63 3.70 3.63
(*b*) On whole milk to which cream was added	9.41	9.57 9.40 9.35 9.35 9.51
(*c*) On skimmed milk	.92	1.02 1.02
Sample thrice skimmed	.42	.40 .32

PARSON'S METHOD OF DETERMINING FAT IN MILK.

[C. L. Parsons, First Annual Report of the Agricultural Experiment Station of New Hampshire, page 72.]

Apparatus.—Three pipettes holding 100, 50 and 25 cubic centimetres; a graduate holding 10 centimetres; a measuring tube about 8 inches in length graduated in tenths of a centimeter from 0 to 5; a few long, slender cylindrical bottles about 11 inches high, 1.5 inches diameter, holding about 250 centimetres; a few small flasks holding about 70 centimeters with the neck cut off obliquely, and of a size to fit easily into the top of the measuring tube, graduated from 0 to 5 centimeters; the measuring tube is expanded slightly at the top to receive the neck of this flask; a drying over 8 inches wide, 12 inches broad, and 14 inches high.

Solutions required.—(1) Gasoline boiling at about 80° to 100° F.; sold usually at 86°, 88°, or 90° gasoline. (2) Caustic soda solution made by dissolving one part caustic soda by weight in two parts of water. (3) 93 per cent. alcohol having one ounce of castile soap dissolved in each gallon. (4) Strong acetic acid.

Directions for analysis.—Place 100 centimetres of the milk into one of the long cylindrical bottles, add to it 10 centimeters of the caustic soda solution and 5 centimeters of the alcoholic soap solution; add then 50 centimeters of the gasoline, shake thoroughly five or six times at intervals for one-half hour; if at the end of this time the gasoline solution does not separate, add another 5 centimeters of the alcohol solution, and turn the bottle slowly over two or three times. Repeat this operation if necessary until four portions of the alcohol have been added or a separation takes place. As soon as a sufficient quantity of the gasoline solution has separated take out 25 centimetres of it, place in one of the small flasks of which the neck has been cut off obliquely, evaporate the gasoline, and add two drops of acetic acid to the residual fat. Place the flask in the drying oven at about 250° for one and one-half hours, after which put the fat into one of the measuring tubes which has been previously placed in the oven. Let the flask remain dripping for ten minutes in the oven, and remove the last drop by drawing the neck of the flask across the neck of the tube. Allow the measuring tube to cool until the fat begins to solidify, and read the number of the centimetres occupied thereby. The percentage of fat is then read from the accompanying table.

ANALYSIS OF SKIM MILK AND BUTTERMILK.

These substances may be treated almost exactly like whole milk. The fat need not be dried more than three-quarters of an hour unless it exceeds 1 per cent.

CREAM.

Cream should first be diluted with an equal volume of water, and then 50 centimetres taken for analysis, and the process carried on as for whole milk, The drying of the fat should continue for about two hours. Care should be taken not to operate the gasoline near a flame, since it ignites very readily, and serious accidents might occur.

USING THE TABLES.

Find the first two figures of the reading obtained from the scale in the left-hand column marked centimetres; then pass horizontally to the right to the column which has the third reading for its heading, and the percentages of fat corresponding to the reading of the scale is found.

For milk, skimmed milk, and buttermilk, containing from 18 *per cent. to* 8.56 *per cent.*

Cubic centi-meters.	0.	1.	2.	3.	4.	5.	6.	7.	8.	9.
0.1	.18	.20	.22	.24	.26	.27	.29	.31	.33	.35
0.2	.36	.38	.40	.42	.44	.45	.47	.49	.51	.53
0.3	.54	.56	.58	.60	.62	.63	.65	.67	.69	.71
0.4	.72	.74	.76	.78	.80	.81	.83	.85	.87	.89
0.5	.90	.92	.94	.96	.98	1.00	1.02	1.04	1.06	1.08
0.6	1.09	1.11	1.13	1.15	1.17	1.18	1.20	1.22	1.24	1.26
0.7	1.28	1.30	1.32	1.34	1.36	1.37	1.39	1.41	1.43	1.45
0.8	1.46	1.48	1.50	1.52	1.54	1.56	1.58	1.60	1.62	1.64
0.9	1.65	1.67	1.69	1.71	1.73	1.75	1.77	1.79	1.81	1.83
1.0	1.85	1.87	1.89	1.91	1.93	1.94	1.96	1.98	2.00	2.02
1.1	2.04	2.06	2.08	2.10	2.12	2.13	2.15	2.17	2.19	2.21
1.2	2.23	2.25	2.27	2.29	2.31	2.33	2.35	2.37	2.39	2.41
1.3	2.43	2.45	2.47	2.49	2.51	2.53	2.55	2.57	2.59	2.61
1.4	2.63	2.65	2.67	2.69	2.71	2.73	2.75	2.77	2.79	2.81
1.5	2.83	2.85	2.87	2.89	2.91	2.93	2.95	2.97	2.99	3.01
1.6	3.03	3.05	3.07	3.09	3.11	3.13	3.15	3.17	3.19	3.21
1.7	3.23	3.25	3.27	3.29	3.31	3.33	3.36	3.38	3.40	3.42
1.8	3.44	3.46	3.48	3.50	3.52	3.54	3.56	3.58	3.60	3.62
1.9	3.64	3.66	3.68	3.70	3.72	3.75	3.77	3.79	3.81	3.83
2.0	3.85	3.87	3.89	3.91	3.93	3.96	3.98	4.00	4.02	4.04
2.1	4.06	4.08	4.10	4.12	4.14	4.17	4.19	4.21	4.23	4.25
2.2	4.27	4.29	4.31	4.33	4.35	4.38	4.40	4.42	4.44	4.46
2.3	4.48	4.50	4.52	4.55	4.57	4.59	4.61	4.64	4.66	4.68
2.4	4.70	4.72	4.74	4.77	4.79	4.81	4.83	4.86	4.88	4.90
2.5	4.92	4.94	4.96	4.99	5.01	5.03	5.05	5.08	5.10	5.12
2.6	5.14	5.16	5.18	5.21	5.23	5.25	5.27	5.30	5.32	5.34
2.7	5.36	5.39	5.41	5.43	5.45	5.47	5.49	5.52	5.54	5.56
2.8	5.59	5.61	5.63	5.66	5.68	5.70	5.72	5.74	5.77	5.79
2.9	5.81	5.83	5.85	5.88	5.90	5.92	5.94	5.97	5.99	6.01
3.0	6.04	6.06	6.08	6.11	6.13	6.15	6.17	6.20	6.22	6.24
3.1	6.27	6.29	6.31	6.34	6.36	6.38	6.40	6.43	6.45	6.47
3.2	6.50	6.52	6.54	6.57	6.59	6.62	6.64	6.66	6.68	6.70
3.3	6.73	6.75	6.77	6.80	6.82	6.85	6.87	6.89	6.92	6.94
3.4	6.97	6.99	7.01	7.04	7.07	7.09	7.12	7.14	7.16	7.18
3.5	7.20	7.22	7.25	7.28	7.31	7.33	7.35	7.38	7.41	7.43
3.6	7.45	7.47	7.49	7.52	7.54	7.57	7.59	7.61	7.64	7.66
3.7	7.69	7.71	7.74	7.76	7.79	7.81	7.83	7.86	7.88	7.91
3.8	7.94	7.96	7.99	8.01	8.03	8.06	8.08	8.11	8.14	8.17
3.9	8.19	8.22	8.25	8.27	8.29	8.31	8.34	8.36	8.38	8.41
4.0	8.43	8.46	8.48	8.51	8.53	8.56	......	.....	....	....

Cream, 15 per cent. to 45.60 per cent.

Cubic centi-meters.	0.	1.	2.	3.	4.	5.	6.	7.	8.	9.
1.9	15.01	15.09	15.18	15.27	15.35	15.43	15.52	15.60	15.69	15.77
2.0	15.86	15.94	16.03	16.11	16.20	16.29	16.37	16.46	16.55	16.64
2.1	16.73	16.81	16.90	16.99	17.08	17.16	17.25	17.34	17.43	17.52
2.2	17.60	17.69	17.78	17.87	17.95	18.04	18.13	18.22	18.31	18.40
2.3	18.48	18.57	18.66	18.75	18.84	18.93	19.01	19.10	19.19	19.29
2.4	19.37	19.46	19.55	19.64	19.73	19.82	19.91	20.00	20.09	20.17
2.5	20.26	20.35	20.45	20.54	20.63	20.72	20.81	20.90	20.99	21.08
2.6	21.17	21.26	21.36	21.45	21.54	21.63	21.72	21.81	21.90	22.00
2.7	22.09	22.18	22.27	22.36	22.45	22.54	22.64	22.73	22.82	22.92
2.8	23.01	23.10	23.20	23.29	23.38	23.47	23.56	23.66	23.75	23.85
2.9	23.94	24.03	24.13	24.22	24.31	24.41	24.50	24.60	24.69	24.79
3.0	24.88	24.98	25.07	25.17	25.26	25.35	25.45	25.54	25.64	25.73
3.1	25.83	25.92	26.01	26.10	26.20	26.29	26.39	26.49	26.59	26.68
3.2	26.77	26.87	26.97	27.07	27.17	27.26	27.36	27.46	27.55	27.65
3.3	27.74	27.84	27.94	28.04	28.13	28.23	28.33	28.42	28.52	28.62
3.4	28.72	28.81	28.91	29.00	29.10	29.20	29.30	29.40	29.50	29.60
3.5	29.70	29.80	29.90	30.00	30.10	30.19	30.29	30.39	30.49	30.59
3.6	30.69	30.79	30.89	30.99	31.09	31.19	31.29	31.39	31.49	31.59
3.7	31.69	31.79	31.89	31.99	32.10	32.20	32.30	32.40	32.50	32.60
3.8	32.71	32.81	32.91	33.01	33.11	33.21	33.31	33.41	33.51	33.61
3.9	33.72	33.82	33.93	34.03	34.13	34.24	34.35	34.45	34.55	34.65
4.0	34.75	34.85	34.96	35.06	35.16	35.27	35.38	35.48	35.59	35.69
4.1	35.80	35.91	36.01	36.11	36.21	36.31	36.41	36.52	36.62	36.73
4.2	36.83	36.94	37.04	37.15	37.25	37.36	37.47	37.57	37.68	37.78
4.3	37.89	38.00	38.10	38.21	38.31	38.42	38.53	38.64	38.74	38.85
4.4	38.96	39.07	39.18	39.28	39.39	39.50	39.61	39.71	39.82	39.93
4.5	40.04	40.15	40.26	40.37	40.48	40.59	40.70	40.81	40.92	41.03
4.6	41.14	41.25	41.36	41.47	41.58	41.69	41.80	41.91	42.02	42.13
4.7	42.24	42.35	42.46	42.57	42.68	42.79	42.90	43.01	43.12	43.23
4.8	43.34	43.45	43.57	43.68	43.79	43.90	44.02	44.13	44.25	44.36
4.9	44.47	44.59	44.70	44.82	44.93	45.04	45.15	45.27	45.38	45.59
5.0	45.60	....	....	....	....	....	....	....	....	....

SHORT'S METHOD OF FAT ESTIMATION IN MILK.

[Reitmaer. Zeitschrift für Angewandte Chemie, 1889, page 288.]

Comparative determinations are given for Soxhlet's areometric and Short's method. In most cases the quantity of fat as determined by Short's method was greater than by the ether process. The milk was bought in open market. Some of the numbers obtained are given below.

Samples in which Short's method gave lower results.

Number.	Fat by Soxhlet.	Fat by Short.
2	2.07	2.05
3	2.24	2.15
4	2.84	2.39
5	3.00	2.69
6	2.19	2.14
7	2.30	2.24

Samples in which Short's method gave higher numbers.

Number.	Fat by Soxhlet.	Fat by Short.
8	2.97	3.09
9	3.04	3.18
10	3.60	4.29
11	2.84	2.95
12	3.33	3.89
13	2.99	3.69
14	3.76	4.48
15	3.76	4.19
16	3.98	4.58
17	1.96	2.39
18	4.46	4.98 4.98 4.98
19	1.65	1.70 1.65 1.60

Triplicates by Short's method agree well together. The cause of the discrepancies in the two sets of operations was not discovered.

THE ANALYSIS OF MILK WITH A VIEW TO DETERMINING ITS ADULTERATIONS.

By Robert Bourcart.

[Bulletin *de la* Société Chimique de Paris, January 5, 1889, page 24.]

The two fraudulent practices which it was sought to detect are the addition of water to the milk and the removal of the cream therefrom. In more than 800 samples examined, no other falsifications have been found.

SPECIFIC GRAVITY OF THE MILK.

For this purpose the lactodensimeter of Quevenne improved by Soxhlet was used. In taking the specific gravity of the milk it is necessary to wait until it attains its maximum contraction, which amounts to from 1 to 1.5° and is caused by the slow solidification of the fatty particles. The time necessary to secure this maximum contraction varies according to the external temperature between twelve and twenty-four hours.

DETERMINATION OF THE PER CENT. OF FATTY MATTER.

The method employed was the areometric method of Soxhlet. The gravimetric determination of Soxhlet was also used.

DETERMINATION OF TOTAL SOLIDS.

Flat platinum dishes weighing about 20 grams were employed for this purpose. Twenty cubic centimeters of milk were employed in each determination.

ESTIMATION OF ASH.

The residue from the determination of total solids is burnt with a small flame, the dish at the end being covered with a watch glass and cooled in a desiccator and weighed. The phosphates and chlorides are estimated volumetrically in the ash.

ESTIMATION OF MILK SUGAR.

The estimation of milk sugar is carried on by means of the alkaline copper solution.

ESTIMATION OF LACTIC ACID.

A few drops of a solution of phenolphthalein are added to the milk, and the titration carried on by means of decinormal soda solution.

METHOD OF CALCULATION OF THE ANALYSES.

One per cent. of solid substances, not counting fatty matter, increases the specific weight of the milk by .004. One per cent. of fatty matter diminishes the specific gravity of the milk by .001, from which the following formulæ are derived:

$$(1)\ P = 1 + 0.004\,(a - g) - 0.001\,g$$

$$(2)\ a = \frac{P - 1 + 0.005}{0.004} - g$$

In the above formulæ, P represents the specific gravity, *a* the total dry substance, and *g* the total fat. In translating the above formulæ the following simple data are obtained:

1. Multiply the per cent. of fatty matter found by 5.
2. Add to it the specific gravity of the milk in degrees of the lactodensimeter.

3. Divide the sum obtained by 4.

You will then have the per cent. of total dry substance in the milk. By the aid of this formulæ one of the three following constituents may be found, the other two being known, namely, the specific gravity, total solids, total fatty matter.

DETERMINATION OF THE REMOVAL OF CREAM FROM MILK.

Every milk which contains less than 3 per cent. of fatty matter may be considered as skimmed, unless it can be proved that the cows furnishing this milk are in some respects abnormal; that is, poorly nourished or sick.

ADDITION OF WATER.

Water can be added to a milk which has a normal content of fat, or on the contrary to a milk too rich or too poor in cream. The specific gravity of a whole milk ought to be at least 1.029. The specific gravity of a skimmed milk which contains about 1 per cent. of fat ought to be equal to 1.0325. If water has been added to the milk a less specific gravity for the skimmed milk than the one given would be found. The total dry matters of a whole milk vary between 12 and 13 per cent. The total dry residue less fat in a milk varies from 8.6 to 9.1 per cent. If water, however, has been added, this residue falls below 8 per cent. The percentage of ash in a whole milk will amount to about .65 per cent., rising as high as .72 per cent. The addition of water will of course diminish the percentage of ash.

QUANTITATIVE DETERMINATION OF THE ADDITION OF WATER TO MILK AND THE REMOVAL OF CREAM THEREFROM.

By Joseph Herz.

[Chem. Zeit. Repert., June 24, 1888, page 175.]

In the exact estimation of the specific gravity and of the percentage of fat, either the quantity of total solids can be determined by Fleischmann's formula

$$t = 1.2 \times f + 2.665 \frac{100 \times S - 100}{S}$$

or the value can be directly determined from a table prepared for this purpose. The method of using this table and for calculating the percentages thereby are described in the twenty-seventh Bericht der chemischen Gesellschaft of Würzburg, 1888, volume 5.

THE SIGNIFICANCE OF THE DIPHENYLAMINE REACTION IN THE EXAMINATION OF MILK.

By Dr. Möslinger.

[Seventh Report of the Bavarian Chemists' Union, page 82.]

The author calls attention to the detection of nitric acid in milk as a means of judging of the addition of water thereto as pointed out by Fuchs and Uffelmann, and also to the researches of Soxhlet on the same subject. The method of Soxhlet is as follows: 100 cubic centimeters of milk are boiled after the addition of 1.5 cubic centimeters of a 20 per cent. chloride of lime solution. A small part of the filtrate is treated with as many drops of concentrated sulphuric acid, in which about 2 per cent. of diphenylamine are dissolved, as are necessary to make the solution turbid. About 2 cubic centimeters of the liquid prepared as described are placed in a test tube over the same quantity of concentrated sulphuric acid. If nitric acid, nitrates, or nitrous acid are present, a ring is formed at the point of union of the two liquids; and this ring appears in from a few minutes to a few hours, according to the quantity of nitric acid present. Much smaller quantities of nitric acid may be detected by the distillation process in the following manner: Four hundred and fifty cubic centimeters of milk are boiled with 6 to 7 cubic centimeters of a 20 per cent. chloride of calcium solution filtered, and the filtrate about 300 cubic centimeters treated with 2 cubic centimeters of concentrated sulphuric acid and about 120 to 150 cubic centimeters distilled off. The distillate is made slightly alkaline with caustic soda and evaporated in a platinum dish to about 5 cubic centimeters, and this residue tested for nitric acid, as described above. It is recommended by Szilasi to apply this reaction directly to the milk by allowing a few drops of milk to flow onto the diphenylamine sulphuric acid solution, which is allowed to remain at rest until the reaction appears. The various modifications of the diphenylamine method are described and discussed on pages 83, 84, 85, 86, and 87. As a result of all the investigations made, it is concluded that the detection of nitric acid by means of diphenylamine in the serum of milk gives a valuable indication for judging whether an adulteration of milk with water has taken place; yet the proof for such an adulteration can not be based exclusively upon the detection of nitric acid, and other grounds of proof must be secured in order to be able to assert that a milk has been watered. The best method for applying the test, as agreed upon by the Association ot Bavarian Chemists, is the following one:

One hundred cubic centimeters of milk after the addition of 1.5 cubic centimeters of 20 per cent. chloride of lime solution are boiled and filtered.

Twenty milligrams diphenylamine are dissolved in 20 cubic centimeters of dilute sulphuric acid (1 to 3), and this solution made up to 100 cubic centimeters with pure concentrated sulphuric acid.

Two cubic centimeters of the diphenylamine solution, as prepared above, are placed in a small porcelain dish. About .5 cubic centimeters of the filtrate of the milk obtained as described above are allowed to flow drop by drop into the midst of the diphenylamine solution, and without mixing allowed to stand for two or three minutes. The dish is then gently waved from side to side and allowed again to stand for a few moments, and this is continued until the presence of nitric acid is indicated by the appearance of the more or less intense blue line, which gradually widens until the whole mass is colored more or less intensely blue.

On pages 88, 89, 90, 91, and 92 of the work cited are found reports of the discussion of the diphenylamine reaction by various members of the Association of Bavarian Chemists.

ESTIMATION OF ALBUMINOIDS IN MILK.

By J. Sebelien.

[Zeitschrift Physiologische Chemie, Volume 13, page 135.]

Milk contains three albuminoids, namely: caseine, lact-albumen, and traces of lacto-globuline. In order to test the method of estimating quantitatively all the albuminoids and determining them separately, a number of experiments was made. These experiments were made first with pure caseine containing small quantities of calcium chloride and sodium phosphate, second with lact-albumen, and third with egg albumen.

An investigation was made of Ritthausen's method of precipitating the total albuminoids with copper sulphate. The total nitrogen in the solution and in the precipitate and in the filtrate was estimated by the Kjeldahl method. It was found when copper sulphate was used that the filtrate was free from nitrogen. When lead acetate was used as the precipitating agent nitrogen was still left in the filtrate. Tannic acid was also found to precipitate completely all the nitrogenous bodies in milk of a proteid nature. Albumoses and peptones are incompletely precipitated by tannic acid. Phospho-molybdic acts much the same as tannin, except that it causes a more complete precipitation of the last two substances mentioned. When milk is saturated with ammonium sulphate all nitrogenous bodies are precipitated except peptone, and if this is present it may be identified in the filtrate by Kühne's reaction, or by the precipitate it gives with tannic acid. No peptones, however, were found in milk. It was also found that albumoses were absent.

The method of estimating the total nitrogen in the precipitate produced by tannic acid and multiplying this by 6.37 is recommended as the most accurate for determining the total quantity of proteids present. For separating the caseine and lact-albumen magnesium sulphate is the best re-agent. It precipitates all the caseine with only a trace of lacto-globuline, and leaves all the lact-albumen in solution. Sodium chloride was also found to precipitate all the caseine. Boiling was found to be a very incomplete method of separating the lact-albumen after the precipitation of the caseine.

ESTIMATION OF CASEINE BY THE METHOD OF KJELDAHL.

By L. L'Hote.

[Comptes Rendus, January 7, 1889, page 61, and April 15, 1889 page 819.]

In determining the nitrogen in caseine by the Kjeldahl method, L'Hote found at first only 9.24 to 9.56 per cent. instead of 13.38 per cent. given by soda lime. When, however, the decomposition was made in a flask with a long neck and with Nordhausen acid, and the digestion continued for thirty-seven hours, the nitrogen obtained was 12.90 per cent. The liquid, however, was still colored. From this it is concluded that the method of Kjeldahl does not present the simplicity, the rapidity, and the certainty which are secured by the soda-lime method for substances like caseine.

DETECTION OF BICARBONATE OF SODA IN MILK.

By L. Pade.

[Journal de Pharmacie et de Chimie, No. 6, September 15, 1889, page 251.]

Bicarbonate of soda is the salt most frequently employed to prevent the formation of coagula in milk. In more than one hundred experiments upon pure milks it has been shown that one drop of a deci-normal solution of sulphuric acid was always sufficient to render acid the ashes from 10 cubic centimeters of milk. It seems, therefore, from this property that taking the alkalinity of the soluble ashes of a milk it might be possible to determine the amount of bicarbonate added, but it was found that on adding 1 gram of bicarbonate to a litre of milk there was found in the soluble ashes only .34 to .40 gram calculated from the alkalinity. This process, therefore, while it is sufficient to determine the presence of bicarbonate of soda, is of no value in determining its quantity. Indeed, during incineration, two thirds of the carbonate employed was transformed into phosphate of soda and carbonate of lime by reacting upon the phosphate of lime contained in the milk. From this reaction the ashes of a milk to which carbonate of soda has been added contains phosphate of soda; but since the soluble ashes of a pure milk are only very feebly alkaline, and contain only traces of phosphoric acid, it is sufficient in order to determine exactly the quantity of bicarbonate of soda added to take the alkalinity of the ashes and to determine the acid which they contain.

To effect these two determinations the following method is employod: 25 cubic centimeters of milk are dried and incinerated, and the alkalinity of the ash is determined by means of a deci-normal solution of sulphuric acid The volume of this acid used, expressed in cubic centimeters, multiplied by .0084 gives the quantity of bicarbonate not transformed into phosphate of soda which the 25 cubic centimeters of the milk contained. Afterwards, by multiplying the volume by .0336 we obtain the percentage. In this neutral solution to which 2 cubic centimeters of slightly acid solution of acetate of soda have been added we determine the phosphoric acid by a standard uranium acetate solution.

The uranium solution is titrated in such a way that 1 cubic centimeter corresponds to .01 gram of bicarbonate of soda for 100 cubic centimeters of milk. The strength of the liquor derived from the transformation of the carbonate of soda into phosphate of soda is seen in the following formula:

$$4\ (CO^3NaH) + (PO^4)^2Ca^3 = 2PO^4Na^2H + 3CO^3Ca + CO^2 + H^2O.$$

According to this formula, 336 grams of bicarbonate of soda give 284 grams of phosphate of soda, or for each gram of bicarbonate, .84523 of phosphate of soda containing .4226 of phosphoric acid.

The uranium solution is titrated volume to volume with a solution containing 3.11 grams of phosphate of soda and ammonia per litre. This weight of phosphate of soda is equivalent to 2.5 grams of bicarbonate of soda. If then the determination is made on 25 cubic centimeters of milk, 1 cubic centimeter of the uranium solution or of the phosphate of soda and ammonia solution is equal to .01 gram of bicarbonate of soda for each 100 cubic centimeters of milk.

Mr. Wiley then read the methods proposed for the analysis of milk for the next year.

Mr. Voorhees asked whether the results obtained by the reporter by the Adams method were entirely satisfactory?

Mr. Wiley said that when the coils could be controlled and carefully extracted and weighed out of contact with the air the results were perfectly satisfactory; otherwise they were not.

It was moved that the recommendations of the reporter covering the method of milk analysis be adopted.

The motion was seconded and carried.

Mr. Wiley continued by reading the alternate methods of milk analysis.

It was moved that the portion of the report last read be accepted and adopted.

The motion was seconded and carried.

Mr. Jenkins said that he understood the reporter to recommend the alternate methods read simply as a check.

Mr. Wiley said that the understanding of Mr. Jenkins was correct.

Mr. Jenkins said that he supposed Mr. Wiley would not use the alternate methods alone for the determination of fat.

Mr. Wiley said that he certainly would not.

The president asked whether all the optional methods given were to be used simply as methods of checking other work.

Mr. Wiley said that the alternate method first presented might be used instead of the analyst's method, but that the other methods given were to be used only as checks.

Mr. Scovell said that that ought to be stated in the report.

Mr. Wiley said that it would be stated in the report as an alternate method.

It was moved that the portion of the report last read be accepted and adopted.

The motion was seconded and carried.

Mr. Wiley said that it was proposed to allow the method for the estimation of sugar in milk to remain exactly as printed in the last Bulletin, no change had been found necessary.

It was moved that the recommendation of the reporter in regard to the estimation of sugar be accepted and adopted.

The motion was seconded and carried.

Mr. Wiley said that in regard to the estimation of total solids he proposed to permit the alternate method of last year with a slight change to stand as the sole method of determining moisture for the coming year. He would say that he was convinced of the slight inaccuracy of this method; he was not yet able, however, to change it.

Mr. Wiley said that the method stated was almost exactly the same as that given last year; the difference being that the method adopted last year prescribed the evaporation "in a watch glass or platinum dish," etc. He would say in regard to this method that it would give perfectly concordant duplicate results, and by its use all would get the same proportion of moisture. His experiments with the coil method, and with drying this in hydrogen had proved to him, however, that the whole of the moisture was not eliminated by this process; that the total solids as indicated by this method were higher than they should be. But this method (drying in hydrogen) was so difficult of manipulation and the other was so simple and so nearly accurate that he recommended it as the sole method of estimating the water.

Mr. Jenkins inquired whether the method of determining the water by drying with sand was included in the scheme.

Mr. Wiley said that it was not included in the scheme of last year. He had practiced the drying with sand, the drying on asbestos, and the drying in a dish with this small quantity, and could not detect any difference in those methods. In using the dish employed by himself a very thin film was formed on the bottom.

Mr. Jenkins said that he had always used the sand method where great accuracy was desired. He found the same result whether he used the sand method or the Adams method, being very careful as to the details of manipulation, but preferred the sand method to the Adams method for the reason that it was so much easier to carry out. He had tried gypsum but the results were not comparable with those given by sand; perhaps the gypsum itself could not be perfectly dried at 100°, and gave off a variable amount of water. He had regarded the sand method as absolutely accurate, having compared the results by that method with the results by drying the milk in hydrogen gas. Some years ago in a series of investigations carried on in Germany, comparing the sand method with the method of drying in hydrogen, which was then supposed to be a nearly perfect method, the total solids by the sand method were found to be less than the tenth of a per cent. higher than by the method with hydrogen. He would be glad to see the sand method as at least a substitute method.

Mr. Wiley said that he was perfectly willing to adopt it as the method.

He thought there ought to be but one and the sand would give the same results according to his experiments. He was willing to say evaporate in a dish about half full of sand. With the permission of the association he would adopt that method, simply drying in a flat-bottom dish containing from ten to fifteen times as much sand as the weight of milk used, which would be 15 or 20 grams of sand.

It was moved that the foregoing recommendation of the reporter be accepted and adopted.

The motion was seconded and carried.

Mr. Wiley continued the reading, giving the method for estimating total nitrogenous matter.

Mr. Wiley said that the only difference between the method reported and that of last year was to evaporate all the milk to dryness, which was very easily accomplished in the digestion flask.

Mr. Jenkins said that he noticed that the factor was not given. He supposed it to be 6.25 but thought it would be well to have it fixed; 6.25 a severybody knew was not the correct factor; he supposed 6.40 would be nearer.

Mr. Wiley said that he had meant 6.25, but as to that the chemists were feeling around in the dark. It was hardly necessary to put in the factor since it was said to be in the "usual way", referring to the standard method adopted for nitrogen or digestion and titration.

It was moved that the recommendation of the reporter last above set forth be accepted and adopted.

The motion was seconded and carried.

Mr. Wiley inquired whether Mr. Jenkins used the spindle of the New York Board of Health in determining the specific gravity of milk.

Mr. Jenkins said that in his laboratory work he did not trust to the use of a spindle preferring the Westphal balance. If he had a large amount of work forced upon him he should use the spindle, but he regarded the balance as a much more accurate method.

It was moved that the scheme of analysis recommended by the reporter on dairy products, as theretofore presented item by item and as modified by the several amendments proposed and made, be adopted as a whole.

The motion was seconded and carried.

The president stated that the report upon fermented liquors was next in order. Mr. Rising, the reporter, was absent but his report had been received and the secretary would read it together with accompanying communications.

Mr. Wiley read the report of the reporter upon fermented liquors as follows:

REPORT UPON FERMENTED LIQUORS.

The methods of wine analysis are largely determined by the motive which suggests the analysis. In general one of the two following reasons may have caused the examination of the wine:

(1) The desirability of determining the presence or absence of adulterations in the

wine; (2) the desirability of comparing any given wine with other wines, either for the sake of determining its type or for comparing the composition of wines of the same type.

The first of these reasons is the one which ordinarily has to be considered, and the methods of analysis are suggested largely for the purpose of determining whether the given sample is adulterated or not.

The following methods are suggested as a guide to chemists in making such examinations: In many of the determinations absolutely accurate results can not be obtained, and consequently the best that we can do is to aim at results which can be compared with those obtained by a large number of chemists.

We have the methods recommended by the commission of Prussia appointed to establish uniform methods for the analysis of wines, and also those adopted by the Bavarian Union.

Where there has been any appreciable difference I have adopted the methods of the Bavarian Union. I believe that by so doing we shall approach an uniform standard throughout the world, and then the results of chemists can be comparable.

GENERAL METHODS OF JUDGING OF THE PURITY OF WINES.

The systematic adulteration of wines is done for a purpose well defined and in very many cases skillfully carried out. A clear understanding of some of the reasons or motions which induce dealers to adulterate wines may be of service to the chemist who tries to detect these adulterations. I shall attempt then to enumerate some of the more important of these reasons.

The use of antiseptics.—There is much wine in various countries which will not keep, or in regard to whose keeping qualities there is grave doubt; wines which will not bear shipment. The producer and wine dealer is at a loss to market them and tries to counteract this tendency by using antiseptics. The fortifying of wines with alcohol is a case in which this practice has become legitimate, and fortified wines are recognized and lawful objects of merchandise.

Not so, however, with other substances of this class. Salicylic acid, sulphurous acid, and sulphites, boracic acid, and whatever other substances may be used for this purpose, careful tests should be made to discover whether any of these are present in the wine. It has already been sufficiently demonstrated that a trace of boracic acid may occur in the pure wine, this will however occur only as a trace, while any appreciable quantity can always be set down to an adulteration. A trace of sulphurous acid may be found in a wine from a cask which has been freely sulphured.

The use of agents to reduce the acidity of wines.—Some base such as carbonate of potash is most frequently used, but this is readily detected in the ash. The amount of soluble alkali in the base is determined by titrations with standard alkali. If mineral matter has been added it can easily and certainly be detected by investigating the ash.

Salt, alum, plaster, etc., are sometimes added to brighten and clarify the wine, and will be found in the mineral matter.

Wines may be stretched; water is added to the wine and then alcohol to bring it up to normal strength. In these wines the relation between the constituents of the wine is disturbed and a comparison with a normal wine will show how changed this relation is, or an attempt may be made to prepare an artificial wine. The discrepancy between the constitution of such a wine and a true one will be still greater.

The addition of sugar (glucose) to grape lees (petiolization) or to the unripe grapes (gallization) are practices more or less employed when the grapes do not ripen well.

The regulation of the Treasury Department for the analysis of foods, drugs, etc., I think might well be followed:

"Wine is the liquid product which results from the alcoholic fermentation of the juice of fresh grapes with such additions only as are essential to the stability or keeping of the liquid.

"The addition of water, distilled spirits, glycerine, sugar, coloring matter, and antiseptics shall be considered adulterations.

"Imitations and artificial wines and demi-wines as hereinafter defined shall also be considered as adulterations.

"Artificial wines are those made without grape juice, with a mixture to imitate wine, such as water, brandy, glycerine, sugar, tartaric acid, œnanthic ether, etc.

"Demi-wines are those resulting from the addition to the must and natural wine of water and other substances intended to produce the taste of wine in such diluted and attenuated liquids (sugar, glycerine, brandy), or made in the same manner with grape marc already used in the preparation of must." (Austrian decree of September 16, 1880.)

"Wines which are made wholly from pure grape juice very seldom contain a less quantity of extract than 1.5 grams in 100 cubic centimeters wine. If wines poorer in extract occur they should be condemned, unless it can be proven that natural wines of the same district and vintage occur with a similar low content of extract.

"After subtracting the 'fixed acids' the remaining extract (*extractrest*) in pure wines, according to previous experience, amounts to at least 1.1 grams in 100 cubic centimeters, and after subtracting the 'free acid,' at least 1 gram. Wines which show less *extractrest* are to be condemned in case it can not be shown that natural wines of the same district and vintage contain as small an *extractrest*.

"A wine which contains appreciably more ash than 10 per cent. of its extract content must contain, correspondingly, more extract than would otherwise be accepted as a minimum limit. In natural wines the relation of ash to extract approaches very closely 1 to 10 parts by weight, still; a considerable deviation from this relation does not entirely justify the conclusion that the wine is adulterated.

"The amount of free tartaric acid in pure wines, according to previous experience, does not exceed one-sixth of the entire 'fixed acids.'

"The relation between alcohol and glycerine can vary in pure wines between 100 parts by weight of alcohol to 7 parts by weight of glycerine, and 100 parts by weight of alcohol to 14 parts by weight of glycerine. In case of wines showing a different glycerine relation an addition of alcohol or glycerine can be inferred.

"As sometimes, during its handling in cellars, small quantities of alcohol (at most 1 per cent. by volume) may find their way into wine, this fact must be borne in mind in judging of its purity.

"These proportions are not always applicable to sweet wines.

"Wines that contain less than 0.14 gram of mineral matter in 100 cubic centimeters are to be condemned, if it can not be shown that natural wines of the same kind and the same vintage, which have been subject to like treatment, have an equally small content of mineral matter.

"Wines which contain more than 0.05 gram of salt in 100 cubic centimeters are to be condemned.

"Wines that contain more than 0.092 gram sulphuric acid (SO_3), corresponding to 0.20 gram potassic sulphate (K_2SO_4), in 100 cubic centimeters, are to be designated as wines containing too much sulphuric acid, either from the use of gypsum or in some other way. (Rules for judging of the purity of wine adopted by the Berlin Commission in 1884, and published August 12, 1884, by the Prussian minister of commerce and trade.)

"Addition of alcohol is to be assumed if the ratio of alcohol to glycerine is greater than 10 to 1 by weight.

"Addition of water and alcohol is recognized by the diminution in the quantity of inorganic matter, especially magnesia, phosphoric acid, and usually potash. Addition of water alone is recognized in the same way.

"Scheelization, *i. e.*, addition of glycerine, is assumed if the ratio of glycerine to alcohol exceeds 1 to 6 by weight.

"The presence of cane sugar is ascertained by a determination of sugar (by Soxhlet or Knapp's method) before and after inversion." (Methods of judging purity of wine adopted by the Union of Bavarian Chemists.)

In regard to beer analysis, I would most respectfully submit the method of the Bavarian chemists as reported one year ago.

Respectfully submitted.

W. B. RISING.

BERKELEY, *September* 2, 1889.

The president stated that the recommendations were nearly the same as those made last year. If no objection was made the report might be considered as adopted.

Mr. Wiley said that he had little to do with wine analysis, but usually objected to a temperature of 15° C.

Mr. Richards said that he would be in favor of making the temperature 25°. He thought that would be nearer the average temperature of the laboratory than 15°.

He moved to amend the recommendation of the reporter by substituting 25° for 15°, as the temperature at which the determinations were to be made.

Mr. Wiley said that the proposed amendment seemed to him to be somewhat on the other extreme; however, 25° was a better temperature than 15°.

Mr. Richards said that he would rather have it a little high than low.

Mr. Wiley said that his average temperature was about 27° in summer, and that in winter he had all temperatures from zero to boiling point, according to the amount of steam turned on. He would like to have the temperature fixed at the point which the chemists found to be their average temperature throughout the year.

Mr. Richards said that his alcohol could be determined very carefully at 25°.

The president said that he believed 25° to be in harmony with the internal revenue tables.

Mr. Gascoyne said that those tables said 15°, for which he was sorry; at least they could be calculated back to that; but he thought 25° a better temperature for work.

The motion made by Mr. Richards was seconded and carried.

Mr. Wiley read the next recommendation of the reporter, as follows:

Alcohol 100 cubic centimeters of wine at 15° distilled (preferably in glass) gave X cubic centimeters alcohol.

Mr. Richards asked whether it was meant that the alcohol or the number of cubic centimeters was unknown?

Mr. Wiley said that he was totally unable to comprehend it.

The president said that on the determination of alcohol the recommendation of the reporter was not clear. It seemed to refer to the determination of the alcohol by distillation, which was the system adopted last year. The question was whether to adopt the recommendation of the reporter or let the method stand as before.

Mr. Scovill said that after reading all of the recommendations the one in question might be explainable.

Mr. Wiley read the next recommendation of the report relating to the "extract."

Mr. Scovell moved that throughout the scheme of analysis, as presented by the reporter, 25° be adopted in substitution for 15°, and that the boiling point of water be substituted for 100°, in order to bring the scheme into accord with the other scheme already adopted.

The motion was seconded and carried.

Mr. Wiley continued the reading of the reporter's recommendations.

Mr. Bowman said that before voting upon the report and recommendations he would like to revert to the change made in the temperature at which the specific gravity was to be taken. He had had almost no experience in this line of work, and it was not apparent that the other members present had any great amount of it to do. Probably, however, some of the members contemplated the undertaking of wine analysis, and it seemed to him that the advantage derivable from working at a more convenient temperature than that prescribed by the reporter would be more than counterbalanced by the fact that their work would be thrown out of line with that of the European chemists. Doubtless Mr. Rising had paid great attention to this subject and had studied it thoroughly, and as 15° being recommended by him as the temperature at which the specific gravity should be taken, and as that appeared to be the temperature at which it was taken by specialists in this line the world over, he moved the reconsideration of the subject.

The motion was seconded.

Mr. Englehardt said that he had taken his picnometer, weighed it and filled it with water of a temperature of 15°, 15°.5, 16° and so on up to 17°, set with distilled water; he had then taken the wine or beer at whatever temperature he had and compared it. Perhaps during the time of the examination, six, eight or nine weeks, the temperature might rise to 20°, 25° or 27° during the summer. A table could be made for reference which would render the method very simple and easy. This he explained.

Mr. Richards said that he understood Mr. Englehardt took the actual density. He asked whether the temperature was compared. Supposing the temperature of wine to be 25°, was that compared with water at 15°?

Mr. Englehardt said that he did not; he compared it at 25°, getting the actual density.

Mr. Richards said that in his picnometers he could always tell the temperature within .5°.

Mr. Englehardt said that he had taken the density of water at each half degree from 15° up to 35°, and that when he had samples of beer or wine to examine he had only to refer to his table for water at that temperature; that saved a great deal of labor and was the shortest and simplest method within his knowledge.

Mr. Richards said that some chemists stated the specific gravity at a certain temperature, but it was impossible to find out with what it was compared. In England 62° was taken by the revenue officers as the standard for all their specific gravities, and water was taken at 62°.

The president said that that was too high.

Mr. Richards said that others took their specific gravities at 15°.5 and compared with water at 15°.5; some took them at 15°.5 and compared with water at 4°, so that it was very confusing and very hard to compare results. On the continent it was taken at 15° and compared with water at 4°; that seemed to be the universal practice. His own results almost always stated the actual density, wine temperature 25° and water at 25° to compare it with.

Mr. Bowman said that if those who were experienced in this line were satisfied that the work would not be thrown out of gear by determining the relative density at 25° temperature he would withdraw his motion.

The president stated that the motion would be considered as withdrawn.

Mr. Richards said that he had lately been engaged in translating the European laws relating to wine and wine adulteration. All the principal countries of Europe had now taken up this subject of adulteration of wine and had made clear definitions of what should be considered a pure wine. In Spain, Italy, Switzerland, Austria, and France pure wine was defined as wine made from the fermented juice of the grape and from nothing else, and addition of any kind, whether of sugar, glucose, or antiseptics, was stated to render the wine artificial, and such wine was to be labeled and sold as artificial wine. The countries referred to now had those laws upon their statute-books and seemed to be enforcing them. It seemed to him some such standard ought to be adopted in this country.

It was moved that the scheme of analysis recommended by the reporter on fermented liquors as theretofore presented, and as modified by the amendments proposed and made, be adopted.

The motion was seconded and carried.

The president stated that the report of the reporter upon the analysis of sugar was next in order. He regretted exceedingly that Mr. Stubbs had sent in no report and was not present at the meeting. One gentleman had traveled hundreds of miles to get the benefit of this report, and when a man failed to present a report he had agreed to make he did great injustice not only to the individuals particularly interested in the special subject, but to the association as a whole. He hoped that in future every man who accepted the position of reporter upon any of these subjects would make it a point either to have a report ready or to notify the executive committee in time to permit the selection of a substitute. Unless there was some objection the question of sugar analysis would be considered as before the convention.

The association would be glad to hear from Mr. Ross, who had a letter from Mr. Stubbs.

Mr. Ross said that he had written Mr. Stubbs about two weeks ago and he did not receive his reply until yesterday. He had informed Mr. Stubbs that he and other chemists had worked upon the subject of sugar analysis during the summer, and had made up some rather interesting data which he would try to have on hand in time for the incorporation in Mr. Stubbs's report at this convention. He understood that Mr. Stubbs had been necessarily absent from Louisiana by reason of illness in his family. Mr. Stubbs now wrote that he was in the midst of sorghum experiments, and that it would be impossible for him to visit Washington at this time. Mr. Stubbs requested him to compile from the data in hand a report for presentation to the association. This he had not yet had an opportunity of doing, but if the association would grant him permission he would make a brief statement of the work carried on by himself, and some others, and would give a few of the results obtained. The report might afterward be written out in such shape that it could be referred to the committee on publications. He did not feel authorized to make any particular recommendations, but would merely present certain of the results.

Mr. Ross made a brief statement of the work referred to. In lieu thereof his full report is here inserted as follows:

NOTES ON SUGAR ANALYSIS.

By D. B. Ross.

With a view to the improvement of the present methods of sugar analysis and the elmination of errors in the processes now employed in the determination of the constituents of cane juices, sirups, molasses, etc., the committee on methods of chemical analysis of the Sugar Chemists' Association of Louisiana, during the past summer made a series of investigations leading to the results herein stated. These investigations were confined almost exclusively to polariscopic methods, especially as regards the determination of sucrose in the presence of invert sugars. As the necessity of double polarization in the analysis of molasses, sirups, and immature sorghum and cane juices is almost universally recognized by chemists conversant with the subject, it was deemed advisable to first make some experiments with reference to the correct expression of the formula used in connection with Clerget's method of inversion. The inaccuracy of the results obtained by the use of Clerget's original constant of 144 having been made apparent in a number of instances, experiments were made to test the correctness of Landolt's revised figure of 142.4.

Though it was found comparatively easy, with the aid of a freezing mixture, to reduce the temperature of the invert solution used to 0° C., yet there was such a condensation of moisture on the glasses of the polariscope tubes that it was impossible to make any readings of the instrument. The difficulty, too, of maintaining the low temperature of 0° C., with an external atmospheric temperature much higher, suggested the necessity of conducting the experiments in an atmosphere approximating in temperature, as nearly as possible, that of the solution under examination. Accordingly, through the courtesy of the New Orleans Cold Storage Company, the committee was permitted to make its investigations in one of their storage rooms, where a constant temperature of about 31° F. was maintained.

The sugar used in the experiments was cane sugar crystallized in unusually perfect rhombic prisms, a normal weight of which, when dissolved in water and made up to 100 cubic centimeters at 17°.5 C., gave a reading of 100, as determined by three independent observers. This normal sucrose solution, however, when reduced to a temperature of 4°C., showed a reading of only 99.7. Fifty cubic centimeters of this solution were next inverted in the manner prescribed by Clerget, hydrochloric acid to the amount of one-tenth of the volume of the solution being added. The temperature of the solution having been lowered to about 2° C., it was placed in a polariscope tube provided with a center tubulure for the insertion of a thermometer. At 1° C. a reading to the left of 42.9 was obtained, which, when corrected for temperature, gave 42.4. At —¼° C. the solution read 42.46—corrected for temperature 42.34; at 0° C. the reading was 42.35; at 4¾° C. the reading showed 42.02—corrected for temperature 42.39. The solution was next raised by a gentle heat to about 15° C., replaced in the tube, and several additional readings made, but the temperature fell so rapidly that no satisfactory duplicate results could be obtained. In these experiments accurately calibrated flasks and tubes were employed, the gaugings were made at 17.5° C., and polariscopic readings were made in duplicate, and in some instances by three independent observers.

It was quite noticeable, too, that at the low temperature employed, the solutions could be read with a clearness and precision unattainable even at the ordinary temperature of the air. The results above given would appear to confirm very fully the correctness of the constant 142.4 as obtained by Landolt. In order to test the effect of the presence of invert sugar, even in comparatively small quantities, upon the direct readings of the polariscope, it was determined to mix varying proportions of a chemically pure invert sugar solution with a given volume of a pure sucrose solution, and to test the solutions thus obtained both by single and double polarization. Samples from the same lot of chemically pure cane sugar were furnished each member of the committee and the subsequent experiments were performed independently in three different laboratories. A normal sucrose solution prepared by dissolving 26.048 grams of sucrose in water and diluting to a bulk of 100 cubic centimeters was first made up and was designated as solution A.

A similar solution was inverted as previously described, and gave the following readings as determined by the respective members of the committee:

	W. P. Martin.	M. Bird.	B. B. Ross.
Readings		—26. at 33. 3°	—27. 4 at 30. 5°
Corrected for temperature.	—42. 5	—42. 65	—42. 65

These results, as corrected for temperature, seem to indicate that the usual compensation of one-half of a degree of the saccharimeter scale for each degree of temperature is a little high though near enough for all practical purposes. A standard invert solution neuturalized with sodium carbonate and made up to 110 cubic centimeters was designated as solution B. Portions of the two solutions were then mixed in the following proportions:

10 cubic centimeters of solution A and 1 cubic centimeter solution B were made up to 100 cubic centimeters forming solution 1.

10 cubic centimeters of solution A and 2 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 2.

10 cubic centimeters of solution A and 3 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 3.

10 cubic centimeters of solution A and 4 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 4.

10 cubic centimeters of solution A and 5 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 5.

10 cubic centimeters of solution A and 6 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 6.

10 cubic centimeters of solution A and 7 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 7.

10 cubic centimeters of solution A and 8 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 8.

10 cubic centimeters of solution A and 9 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 9.

10 cubic centimeters of solution A and 10 cubic centimeters solution B were made up to 100 cubic centimeters forming solution 10.

The solutions thus obtained were subjected to polariscopic tests, the direct reading in two of the laboratories being made in 400 millimeter tubes, with a double compensating Schmidt and Haensch instrument. The temperatures of the solutions at the time of reading were carefully noted and are given below.

Results of single polarization.

	Martin.		Bird.		Ross.	
Solution 1	9. 80	at 30° C.	9. 80	at 33° C.	9. 75	at 29. 5°. C
Solution 2	9. 60	29. 5° C.	9. 50	33°	9. 55	30°
Solution 3	9. 30	29. 5° C.	9. 30	33°	9. 35	30. 5°
Solution 4	9. 20	30° C.	9. 10	33°	9. 10	30°
Solution 5	8. 80	29. 5° C.	8. 90	33°	8. 85	29°
Solution 6	8. 60	29. 5°	8. 70	33° C.	8. 60	29. 5°
Solution 7	8. 40	30°	8. 50	33° C.	8. 35	29. 5°
Solution 8	8. 20	30°	8. 20	33°	8. 10	29. 5°
Solution 9	7. 90	30°	8. 00	33°	7. 85	29. 0°
Solution 10	7. 60	30°	7. 70	33°	7. 60	39. 5°

From the above results it will be seen that for each cubic centimeter of the invert solution B mixed with 10 cubic centimeters of the sucrose solution A, there has been an average decrease in the apparent per cent. of sucrose of .24 at an average temperature of 29.8° C, .23 at an average temperature of 33°, and .24 at a temperature of 29.6°. (The quantity of invert sugar in 1 cubic centimeter of solution B, when diluted to 100 cubic centimeters gives an equivalent of .957 per cent. of invert sugar). The normal sucrose solution when inverted and made up to 110 cubic centimeters gives a reading in a 200 millimeter tube of 38.5 at 0°C $\left(\frac{42.4}{1.1}=38.5\right)$; at 29.8° C the reading would be

$$\frac{42.4-\frac{29.8}{2}}{1.1}=25;$$

at 33° C the reading would be

$$\frac{42.4-\frac{33}{2}}{1.1}=23.55;$$

at 29.6° the reading would be

$$\frac{42.4-\frac{29.6}{2}}{1.1}=25.1.$$

Accordingly, 1 cubic centimeter of such a solution raised to 100 cubic centimeters should give a reading to the left at 29.8°, of .25; at 33° a reading of .2355; at 29.6° of .251, whereas the experiments indicate that the right handed readings show a deviation to the left of .24, .23, and .24 for each cubic centimeter of the invert solution present, a comparatively close approximation of the practical to the theoretical results.

In two of the laboratories double polarization tests were made with the following results:

Calculated readings after inversions.

	Bird.	Ross with the use of the constant—	
		142.4	144.
Solution 1	10.07	10.07	9.95
Solution 2	9.91	10.08	9.96
Solution 3	9.95	10.08	9.96
Solution 4	9.96	10.05	9.92
Solution 5	9.99	10.08	9.96
Solution 6	10.03	10.05	9.92
Solution 7	10.01	10.03	9.90
Solution 8	10.04	10.01	9.88
Solution 9	10.08	9.99	9.87
Solution 10	10.12	10.05	9.92

The results given in the second and third columns are obtained by the use of the formulæ

$$R=\frac{100S}{142.4-\frac{1}{2}T} \text{ and } R=\frac{100S}{144-\frac{1}{2}T}$$

respectively.

The average variation from the theoretical percentage with the use of the constant 142.4 was .051 per cent.; while with the figure 144 the variation was .076 per cent. Had 400 millimeter tubes been employed for the invert solutions, as well as in the single polarization tests, the discrepancies would have, without doubt, been much smaller.

In addition to the polariscopic determinations given above, the actual amounts by weight of invert sugar in the solutions examined were determined by means of Fehling's solution. The following are the results obtained, together with the theoretical amounts of invert sugar contained in the solution:

	Theoretical amounts.	Amounts obtained by use of Fehling's solution.	
		Bird.	Ross.
	Gram.	*Gram.*	*Gram.*
Solution 1	.2492	.253	.251
Solution 2	.4984	.506	.5074
Solution 3	.7476	.7438	.7445
Solution 4	.9968	.9918	.9950
Solution 5	1.246	1.2346	1.244
Solution 6	1.4952	1.5031	1.4854
Solution 7	1.7444	1.7659	1.733
Solution 8	1.9936	2.004	1.952
Solution 9	2.2428	2.2673	2.227
Solution 10	2.492	2.530	2.4697

The alkaline cupric liquor used in the above determinations was Violette's modification of Fehling's solution and was standardized by means of a solution of C. P. anhydrous glucose. It will be seen by reference to the above table that approximately one-fourth gram, or multiple thereof, of invert sugar was present in each of the solutions examined, and that for each one-fourth gram contained in 100 cubic centimeters of the solutions, there was an average laevo-rotary effect of 0.24, 0.23, and 0.24 at the respective temperatures of 29.8°, 33°, and 29.6°. At lower temperatures it is readily apparent that this influence would be much greater. The results of these experiments, therefore, seem to indicate the necessity of making due compensation in sucrose determinations for the presence of invert sugar even in small proportions.

Mr. Wiley asked what instrument Mr. Ross used in the investigations.

Mr. Ross said that he used the Schmidt and Haensch double compensating 400 millimeter length of tubes, but that for the reading of invert solutions he used a 220 simply because he had no 400 to use.

Mr. Wiley said that he had been much interested in the report of Mr. Ross and that there was little doubt in his mind that in all solutions inverted by hydrochloric acid 142.5 or something like that was the correct factor to use. Before setting aside the factor 144, however, some different method of inverting the sugar must be had, using a less powerful acid; until such a method were had it could not be decided that a pure solution inverted without destruction of levulose would read 142.5 instead of 144. Such investigations as these were just what were needed. He now uniformly used the factor 142.5 when he inverted with hydrochloric acid of strength recommended, and doubtless got much truer results than when he used the factor 144. He had made numbers of observations on this subject and with most conflicting results.

There were many matters which could have been profitably considered at this meeting, and perhaps some changes might have been adopted or some new method of sugar analysis. Of course it would be useless to attempt to agree upon any method for adoption at this meeting. The matters spoken of could only be mentioned for the information of those who were working, in order that they might be observing and report upon them at another time. He would like while he had the floor to call attention to the instrument he had lately imported for analytical work, and believed that many of those who were working on sugar would wish to get something of the same kind. The instrument was manufactured by Schmidt and Haensch, but only to order. Only two of them had been made. The instrument was devised by Professors Landolt and Lippich; its cost is $350, free of duty. It varied from the ordinary polariscope in having no graduations for cane sugar; giving only angular solution. Its mechanism permitted a most accurate measurement of angular solution; but it was an instrument better fitted for research work than for general analysis.

He proposed this year to review the data of double polarizations on both cane juice and sorghum juice. His old results he was obliged to say were at least in suspicion in regard to the invert sugar. He believed that there was optically inactive sugar in cane juice. It appeared to him that a point was now reached where some of the most interesting discoveries in regard to the nature of these sugars were to be made. It was really necessary to understand the nature of every substance optically inactive in cane juices. He had been asked by the Secretary of the Treasury to prepare a schedule of regulations for the United States customs officers in the examination of sugars, and should do so as soon as possible. Whatever observations were made and regulations prescribed would be presented in connection with the report on sugar at the next meeting. Of course it was necessary to go slowly in this; at the present time the method of sugar examination in the custom-houses was very much in need of revision. No account whatever was taken of invert sugar and the implements employed were open to very grave suspicion.

Mr. Jenkins asked whether it was proposed to include a schedule for the examination of molasses.

Mr. Wiley said that it was.

Mr. Jenkins said that in Connecticut they were particularly interested in molasses, and he had been obliged to make examinations and to appear in court as a witness.

Mr. Warder said that if the discussion on sugar was closed, and if he might be allowed to revert to phosphoric acid, he would be glad to have the attention of the association upon that subject.

Mr. Wiley moved that Mr. Warder be allowed to read his paper upon phosphoric acid.

The motion was seconded and carried.

Mr. Warder read the paper referred to as follows:

REMARKS ON REVERTED PHOSPHORIC ACID.

By Robert B. Warder.

For several years an effort has been made to perfect the recognized method of estimation where it is possible that sweeping changes would lead to a more accurate process. The official method of determining "reverted phosphoric acid" suffers from these two sources of inaccuracy.

(1) So small a quantity must be taken that it is often impossible to secure a fair sample without grinding, and differences in the amount of grinding result in wide discrepancy, especially in the examination of bone.

(2) After the solution of ammonium citrate has acted for a specified time, the solvent action is continued during the very uncertain period required for filtering and washing; hence the total time (and, consequently, the total quantity dissolved) is subject to irregular fluctuations.

In seeking to avoid these disadvantages by a new process the use of other solvents may well be considered. Perhaps dilute citric acid (tried and abandoned some years ago) or some weak volatile acid may be found desirable. I venture to suggest the following, as guiding principles in seeking another method.

(1) The quantity of commercial fertilizer taken should be large enough to secure a fair sample without any grinding by the analyst.

(2) The use of a lower temperature may avoid (among other difficulties) the inconvenience of digestion under pressure.

(3) If a longer time is allowed for the action of the solvent, an error of one minute in the actual time will result in a smaller percentage error in the result.

(4) Instead of using the washed residue it may be desirable to analyse an aliquot part of the solution (which may be quickly decanted upon a filter) to avoid the irregularities in time required for washing.

(5) Details should be so adjusted as to yield results approximately equivalent to those now obtained.

(6) If an improved process is found to yield more constant results, due notice should be given to the manufacturers before its general adoption for fertilizer control.

In order to avoid needless delay, it is suggested that all chemists interested be invited to communicate with an appropriate committee upon the following points:

(1) What is the minimum quantity of a coarse fertilizer that can be taken with safety as a fair sample for analysis?

(2) What re-agent or re-agents are likely to be less inconvenient than ammonium citrate?

(3) What temperature is most convenient?

Mr. Jenkins said that doubtless all present were conscious of a debt of gratitude to the acting Secretary of Agriculture and to others connected with the Department who had made the meeting so pleasant for the members. He moved that a committee of one be appointed to draft and present a resolution expressive of the meeting in this regard.

The motion was seconded and carried.

The president appointed Mr. Jenkins as such committee.

The president stated that the regular business meeting of the convention was next in order. It was provided under the constitution that the election of officers should take place by ballot without nomination.

The president appointed as tellers for the election Messrs. Harrington and Robinson.

The balloting was then proceeded with and the following officers elected:

President, Mr. M. A. Scovell.

Vice-president, Mr. G. C. Caldwell.

Secretary, Mr. H. W. Wiley.

Executive Committee, Messrs. E. H. Jenkins and John A. Myers.

Mr. Jenkins presented the report of the committee on resolutions as follows:

Resolved, That the Association of Official Agricultural Chemists desires to express to the honorable Acting Secretary of Agriculture its appreciation of the interest he had expressed in the objects of this association and of the courtesy and invaluable help he has extended to it in providing a place of meeting and in his generous offer to publish and distribute the report of the proceedings.

It was moved that the foregoing report be accepted and adopted.

The motion was seconded and carried.

The president said that before adjourning he desired, as retiring president, to express to the association his sincere thanks for the honor it had conferred upon him and for the uniform courtesy he had enjoyed at its hands. It was extremely difficult for a man in the chairman's position to preside over a body that was disorderly or disposed to be annoying in any way. Nothing had contributed more to his pleasure during the meeting than had the manifestation of a uniform high sense of precision and earnestness of the work upon which the association was engaged. It had been the most earnest meeting of the association that he had ever attended. He had tried to treat all with perfect fairness in his rulings and to give every one ample opportunity to discuss any measure presented. If any one had felt aggrieved by any of his rulings he trusted that it would be appreciated that he had intended to act for the good of the association and that any such ruling was not due to personal feelings of his own. He thanked the members of the association for their kindness, and especially thanked Mr. Wiley for the assistance he had rendered and for the kindness he had shown in securing so many favors for the association.

Mr. Gaines said that before the meeting adjourned he would like to get an expression of opinion as to the best time for holding these meetings hereafter. They had usually been held in the month of August, which time was inconvenient to some of the members, and he for one would like to see the time set as late in the year as possible, certainly not earlier than September; he thought that October or November would be better.

Mr. Frear said that it would be very pleasant to him personally if the meetings could be held at about the same time as that of the Association of Agricultural Colleges and Experiment Stations. He was not at all prepared to advocate any organic relation with that association, but thought it would be advantageous to many of the chemists, saving time and money if these meetings could be held, at least in every other year, at the same time as those of the association referred to. That association, he believed, contemplated meeting every other year at Washington and the alternate year at some one of the experiment stations. He would like to urge this matter upon the executive committee for serious consideration.

The president stated that the executive committee had considered this subject very carefully each year and that it had been discussed at previous meetings, but the country was so large and the diversity of interests so great that it was impossible to find any time that would suit all the members of the association. The meetings had been held in the middle of winter as well as in the month of August. It appeared that the latter part of August or the early part of September suited every one much nearer than did any other time in the year. The question of the time of meeting was one of the matters left to the executive committee and it was only necessary for the members to express their respective sentiments to that committee. There was nearly always more or less correspondence in reference to this point, and he had no doubt that the executive committee would give the most careful consideration to every suggestion that should be made. The committee had the interests of the association at heart and would endeavor to appoint such a time for the meeting as would accommodate the most of the members.

Mr. Voorhees said that he would like to have an expression of opinion from the members present regarding the time at which the samples should be sent out by the different reporters. He had been unable to do the work this year owing to the fact that the samples were not received at a time when he could attend to them.

Mr. Frear said that he would rather receive the samples from the several reporters at different times so that he could devote attention successively to the various subjects of analysis. He would like to have the fertilizer samples come in some time during his fertilizer season, which extended from the present time until the first of December and from the latter part of March onward.

The president said that the reporters would probably find it to their advantage also to consult the wishes of the members in reference to the time of sending out the samples. It was desired to get reports from as many chemists as possible, and by consulting the wishes of the members in that respect a large number could be induced to make analyses.

Upon motion the convention was then adjourned.

OFFICERS AND REPORTERS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS OF THE UNITED STATES FOR 1889-1890.

PRESIDENT.

Mr. M. A. SCOVELL, Director of the Kentucky Agricultural Experiment Station.

VICE-PRESIDENT.

Mr. G. C. CALDWELL, Chemist of the New York Agricultural Experiment Station.

SECRETARY.

Mr. H. W. WILEY, Chief Chemist, U. S. Department of Agriculture.

EXECUTIVE COMMITTEE.

Mr. E. H. JENKINS, of New Haven, Conn.
Mr. J. A. MYERS, of Morgantown, W. Va.

REPORTERS.

Phosphoric acid.—Mr. W. B. Burney, of Columbia, S. C.

Potash.—Mr. N. T. Lupton, of Auburn, Ala.

Nitrogen.—Mr. E. B. Voorhees, of New Brunswick, N. J.

Cattle foods.—Mr. G. C. Caldwell, of Ithaca, N. Y.

Dairy products.—Mr. E. H. Jenkins, of New Haven, Conn.

Fermented liquors.—Mr. W. B. Rising, of Berkeley, Cal.

Sugar.—Mr. H. W. Wiley, of Washington, D. C.

CONSTITUTION OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.

(1) This association shall be known as the Association of Official Agricultural Chemists in the United States. The objects shall be (1) to secure uniformity and accuracy in the methods, results, and modes of statements of analysis of fertilizers, soils, cattle foods, dairy products, and other materials connected with agricultural industry; (2) to afford opportunity for the discussion of matters of interest to agricultural chemists.

(2) Analytical chemists connected with the United States Department of Agriculture, or with any State or national agricultural experiment station or agricultural college, or with any State or national institution or body charged with official control of the materials named in section 1, shall alone be eligible to membership, and one such representative for each of these institutions or boards, when properly accredited, shall be entitled to a vote in the association. Only such chemists as are connected with institutions exercising official fertilizer control shall vote on questions involving methods of analyzing fertilizers. Any person eligible to membership may become a member at any meeting of the association by presenting proper credentials and signing this constitution. All members of the association who lose their right to such membership by retiring from positions indicated as requisite for membership shall be entitled to become honorary members and to all privileges of membership save the right to hold office and vote. All analytical chemists and others interested in the objects of the association may attend its meetings and take part in its discussions, but shall have no vote in the association.

(3) The officers of the association shall consist of a president, vice-president, and secretary, who shall also officiate as treasurer; and these officers, together with two other members to be elected by the association, shall constitute the executive committee. When any officer ceases to be a member by reason of withdrawing from a department or board whose members are eligible to membership, his office shall be considered vacant, and a successor may be appointed by the executive committee to continue in office till the annual meeting next following.

(4) There shall be appointed by the president, at the regular annual meeting, a reporter for each of the subjects to be considered by the association.

It shall be the duty of these reporters to prepare and distribute samples and standard reagents to members of the association and others desiring the same; to furnish blanks for tabulating analyses, and to present at the annual meeting the results of work done, discussion thereof, and recommendations of methods to be followed.

(5) The special duties of the officers of the association shall be further defined, when necessary, by the executive committee.

(6) The annual meeting of this association shall be held at such place as shall be decided by the association, and at such time as shall be decided by the executive committee, and announced at least three months before the time of meeting.

(7) Special meetings shall be called by the executive committee when in its judgment it shall be necessary, or on the written request of five members; and at any meeting, regular or special, seven enrolled members entitled to vote shall constitute a quorum for the transaction of business.

(8) The executive committee shall confer with the official boards represented with reference to the payment of expenses connected with the meetings and publication of the proceedings of the association.

(9) All proposed alterations or amendments to this constitution shall be referred to a select committee of three at a regular meeting, and after report from such committee may be adopted by a vote of two-thirds of the members present and entitled to vote.

OFFICIAL METHODS OF ANALYSIS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR 1887-'88.

METHODS FOR SUGAR ANALYSIS.

[The association not having adopted any method of sugar analysis, the following processes adopted by the Louisiana Sugar Association were submitted for provisional use.—H. W. W.]

APPARATUS, CHEMICALS, AND CHEMICAL REAGENTS ESSENTIAL TO THE EQUIPMENT OF A SUGAR HOUSE LABORATORY.

Apparatus.—Polariscope with observation tubes, polariscope lamp, funnels, beakers, sugar flasks graduated 100 to 110 cubic centimeters, and smaller size flasks graduated to 50 and 55 cubic centimeters, burettes (with .10 cubic centimeters graduations) and burette support, casseroles, porcelain evaporating dishes, water bath, retort stand, centigrade thermometer, set of Brix hydrometers with cylinder, liter flask, wash bottle, pipettes, glass tubing, filter paper, litmus test paper, pair of balances, set of weights, German-silver sugar dish, with counterpoise and normal sucrose weights, an alcohol lamp for heating purposes, or a Bunsen burner if a gas supply can be obtained.

Chemicals and reagents.—Lead acetate, litharge, copper sulphate, Rochelle salt, caustic soda, potassium ferrocyanide, C. P., anhydrous glucose, acetic acid, hydrochloric acid, sodium carbonate, boneblack, finely cut asbestos, solution basic lead acetate, Fehling's copper solution.

Selection and adjustment of instruments, apparatus, etc.—For all the ordinary analytical work in the sugar-house the polariscopes with the horizontal sugar scale of 100 parts are to be preferred to those in which the circular scale of degrees is employed, and in which the exact angle of rotation and not the direct per cent. of sugar is read. The instruments of this latter description are invariably constructed *a penombre* and require the use of a mono-chromatic (sodium) light in a dark room, and a consequent expenditure of more time and manipulation than in corresponding determinations with saccharimeters adapted to ordinary lamp-light. Of the various makes and styles of instruments of the last-named class, a large proportion have the same normal sucrose weight of 26.048 grams, with a normal length of 200 millimeters for observation tubes. The shadow polariscope adapted to white light is the form of instrument more generally used at present, but the color instruments provided with a regulator for changing the tints of the field of the instruments is frequently preferred by those who experience no difficulty in detecting slight shades of difference in color. The Schmidt & Haensch shadow instrument, with double compensating wedges, and with a capacity for 400 millimeter tubes is to be especially commended for its ease of manipulation and the accuracy of results, it being possible to check each individual reading four times by means of the duplicate scales, while any incidental errors are reduced one-half by the use of the 400 millimeter tubes. The Soleil-Ventzke and Scheibler instruments, and most especially the shadow instruments of the latter make, are extremely well adapted to the ordinary work of the sugar-house laboratory, and in the hands of a careful operator give result whose accuracy may be confidently relied upon.

In the adjustment of polariscopes care should be taken that the sugar used in the preparation of the normal sucrose solution is of absolute purity. Either pure white rock-candy in well-defined crystals, or the best loaf-sugar, extracted with 85 per cent. alcohol and well dried at a temperature slightly below 100° C. should be used. The rock-candy, should be thoroughly pulverized and pressed between sheets of blotting or filter paper previous to weighing, or if the atmosphere is heavily charged with moisture, it will be best to dry it at a temperature just below the boiling point of water.

The normal weight, 26.048 grams, of sucrose is weighed up, dissolved in pure distilled water, and diluted to a bulk of 100 cubic centimeters. The gauging of the sucrose solution should be performed as nearly as possible at 17.5° C., as in sugar analysis that is the standard temperature for measurements and polariscope readings.

For ascertaining the correctness of the zero point of the instrument, the polariscope tube is filled with pure distilled water, and having obtained a uniform tint or shade in the field of the polariscope, the variation in the position of the two zeros is corrected by means of the key applied near the left hand extremity of the vernier scale. Having made this adjustment, the normal sucrose solution should give a reading of 100 if the analyzer and polarizer are in right position with regard to each other.. If, however, no uniformity of shade or tint on both sides of the center line is observed for any position of the movable quartz wedge the analyzer and polarizer are out of adjustment with reference to each other. In order to effect correct adjustment of the parts the movable quartz wedge must be taken out by turning the milled head screw underneath until it can be easily slipped out; the stationary quartz wedge is next displaced by removing the screws which hold it in position and which are usually found almost underneath the screws which hold in place the apparatus for reading the scale. The compensation quartz plate is next removed by opening the cover of the chamber for the reception of observation tubes and unscrewing the plate at the end nearest the observer. The cover having been closed, an observation is taken, and the position of the analyzer is corrected by applying the polariscope key to the screw head on the right of the instrument and just in the rear of the compensation plates. The key is turned until there is a uniformity of shade on both sides of the vertical center line of the field of the apparatus. The compensation wedges and plates having been replaced a reading with an empty tube is made and any deviation in the position of the two zeros is corrected as before explained.

The linear measure of all polariscope tubes should be carefully taken, as any variation from the standard length will cause directly proportional errors in results. This measurement, from end to end, should be effected by means of a pair of dividers or calipers, the divergence of the points of the instrument when applied to a finely graduated metric scale giving accurately the length of the tube. Tubes showing a perceptible variation from the normal length should be rejected or else all readings made in them should be corrected in direct proportion to the error in length. Where instruments have a capacity for 400 millimeter tubes, the long tubes should be invariably used for juices and sirups when practicable, and the 100 millimeter tubes should only be employed in polarization of dark or difficultly decolorizable solutions, as decrease in length of polariscope tubes is attended with a proportional increase in errors. For double polarizations it is far preferable to use those tubes provided with a central tubulure at right angles to their length for the insertion of a thermometer and the recording of temperature at the instant of reading It is also preferred that the tubes be one-tenth longer than those used for direct readings in order to compensate for the dilution of the solution. The Scheibler instruments generally employed, however, will not accommodate tubes of a greater length than 200 millimeters.

In filling tubes with solutions for analysis, it is always best to first rinse them out with the juice or sugar solution under examination, and afterward fill the tube (holding it in a vertical position) until the liquid reaches the upper end and assumes a convex surface; the glass disk is then slipped on horizontally, and the screw cap is adjusted and tightened, absence of bubbles being thus almost invariably insured. The glass covers of the tube should, after filling, also be wiped free of any particles or drops of liquid, silk, chamois skin, or filter paper being used for this purpose, instead of cotton or linen, small particles of which latter substances frequently attach themselves to the glasses. The tubes should be emptied of their contents as soon as the observation is completed, the retention in metallic tubes of solutions, inverted by means of acid, being especially injurious.

The lamp best adapted to polariscope work is of the "German student" pattern, supplied with a clay cylinder with side aperture, oil being preferred to gas as an illuminating agent. The end of the instrument should not be placed nearer than from 2 to 2½ inches from the source of light, as the heat from the lamp tends to affect the cement used in holding the prisms in position.

In order to test the correctness of the calibration of measuring flasks used in the various operations herein described, it will be necessary to first dry the empty flask and then weigh it. Distilled water (whose specific gravity, corrected for temperature, has just been determined) is run into the flask until the lower edge of the meniscus at the surface of the liquid is on a line with the mark on the neck of the flask. The flask and contents are then weighed, the increase in weight divided by the specific gravity of water at that temperature, giving the correct capacity of the flask in centimeters.

Determination of sucrose.—In weighing, the balances used need only be sensitive to within 10 milligrams, as that amount of variation in the weight of a sample would scarcely be appreciable in the readings of the polariscope. Transfer, by means of a pipette, to the tared German silver sugar dish the normal weight of the juice or sirup to be analyzed, a spatula or other device being used in case the sample is not of a liquid consistency.

In the case of juices and thin sirups, the contents of the dish are at once washed into the 100 cubic centimeter flask; sugars, masse cuite, heavy molasses, etc., however, are best dissolved in the dish previous to being transferred.

The solution in the flask is made up to 80 or 90 cubic centimeters, and the least amount of basic lead acetate solution, sufficient to decolorize the contents, is added. The solution is then made up to the 100 cubic centimeter mark and thoroughly shaken before filtering. In case much foaming and bubbling takes place, rendering gauging difficult, the addition of a few drops of ether is found advantageous. The solution is then poured upon a dry filter and the filtrate is collected in a dry beaker, the first portions to pass through being rejected. If it is found impossible to obtain a perfectly clear filtrate, it may be necessary to decolorize with dry powdered boneblack, either mixed with the liquid itself or placed upon the filter. Should it be desired to remove the excess of lead in the solution before polarizing, it can be precipitated out before filtering by means of sodium carbonate or sulphate (preferably the latter). The tube is filled with the clear solution as before described, and the process of reading the instrument is conducted as usual.

Double polarization.—Fifty cubic centimetres of the decolorized solution used in the direct polarization test is placed in a sugar flask, graduated to 50 to 55 cubic centimeters, and hydrochloric acid of specific gravity 1.18 to 1.20 is added until the liquid reaches the 55 cubic centimeter mark, the contents being well shaken. The flask and contents are then heated in a water bath at a temperature of 68° to 75° C. for about fifteen minutes. It is then allowed to cool, freed from the precipitate of lead chloride by filtration, the filtered solution being transferred to a tube of the form previously referred to as being especially adapted to the reading of invert solutions. The reading of the solution to the left is noted, and at the same time its temperature is carefully taken by means of a thermometer inserted in the central tubulure of the tube. In calculating the true per cent. of sucrose from the data thus obtained, instead of Clerget's original constant of 144, the revised figure of 142.4 should be used, as the and committee's experiments show conclusively that 26.048 grams of sucrose, inverted, made up to 100 cubic centimeters gives a reading to the left of 42.4 at 0° C. The correct expression of the formula used in the calculations will then be $R=\frac{100\ S}{142.4-\frac{1}{2}T}$ in which S = the sum of the right and left handed readings of the solution; T = the temperature of the solution at the instant the left handed reading is taken, while R is equal to the corrected per cent. of sucrose. Where it is preferred, the simple proportion $142.4-\frac{1}{2}T : 100 :: S : R$ can be used.

In double polarization tests the readings of the invert solutions should invariably be conducted at, as nearly as possible, the same temperatures as the direct readings.

Specific gravity and density.—Density of juices, sirups, etc., is most conveniently determined in the sugar-house by means of Beaume's or Brix's hydrometer or areometer, preferably with the latter, as the graduations of the scale give close approximations to the percentages of total solids. The Brix spindle should be graduated to tenths, the divisions of the scale being of sufficient length to enable the analyst to note exactly the point to which the stem sinks in the liquid. It is therefore desirable, for accuracy, that the range of degrees recorded by each individual spindle be as limited as possible, this end being best secured by the employment of sets, consisting of not less than three spindles. The solutions should be, as nearly as possible, of the same temperature as the air at the time of reading, and if the variation from the standard temperature of the graduation of the spindle amounts to more than 1° C., compensation therefor must be made by reference to the table of corrections for temperature on page 114, Tucker's Sugar Analysis.

Care should be observed, in taking the density of a juice, to allow it to stand in the cylinder until all air-bubbles have escaped, before reading the spindle. When it is desired to accurately determine the specific gravity of the sample, a specific gravity bottle or picnometer, of 50 cubic centimeters capacity, is employed, and the weight of the amount required to fill the bottle at 15°.5 C, divided by 50, gives its correct specific gravity. When the temperature varies from the standard temperature for specific gravities, the weight of the juice must be compared with an equal volume of water, weighed at the same temperature.

Total solids.—The per cent. of total solids can be approximately ascertained directly from the reading of the Brix hydrometer, or knowing the reading of the Beaume spindle, or the specific gravity of the sample, by reference to the table on pages 116-118, Tucker's Sugar Analysis, the desired percentage can be obtained.

In order to accurately estimate the proportion of total solids in a juice or sirup, from one to three grams (according to the density of the liquid) are run into a porcelain dish or glass schælchen, containing about two grams of finely powdered, ignited silica. The whole is then placed on a water bath and heated until the contents are apparently dry, after which it is transferred to an air bath and dried to a constant weight at not exceeding 110° C. The weight of the dish and silica subtracted from weight of dish and contents after drying, gives total solids.

Total solids may also be determined by Dr. Wiley's method of saturating a dried and weiged piece of filter paper (about 12 by 2 inches in size) with the sample of juice or molasses, and drying at 100° and re-weighing.

Determination of glucose.—In the analysis of juices, 10 grams of the sample are weighed up, transferred to a graduated flask, and made up to 100 cubic centimeters; in estimations of glucose in molasses and sirups, 5 grams are weighed up and diluted to 500 or 1,000 cubic centimeters, according as the proportion of glucose present is relatively small or large.

It is best that the diluted solution contain not more than 2 milligrams of glucose to the centimeter of liquid. Ten cubic centimeters of Fehling's solution are next run into a casserole from a burette and three or four times its bulk of water added; it is then placed over a flame and brought to a brisk boil, care being taken to observe whether or not any of the copper is precipitated as suboxide. If no precipitation is noticed, a few cubic centimeters of the solution under examination are added from a burette and the contents of the casserole are again boiled, stirred well, removed from the lamp, and the precipitate allowed to subside. The process is thus continued, the sugar solution being run in at short intervals and in small quantities at a time, the liquid in the casserole being boiled and allowed to settle each time. When the blue tint has almost disappeared from the liquid, and the dull red of the precipitate has changed to a scarlet, the solution is added very cautiously, stirring and settling as before. When the precipitate subsides readily and the supernatant liquid becomes clear and almost colorless, a test is made to ascertain whether or not all the copper is precipitated. A few drops of the liquid, when filtered and placed on a porcelain casserole cover or crucible top, will, when acidulated with acetic acid, give a reddish brown precipitate with potassium ferrocyanide, if any copper remains in solution. The filtration of a small portion of the solution for testing is best effected by means of Wiley's tubes—easily prepared by making a flange on one end of a piece of glass tubing, about ⅜ inch diameter and 5 or 6 inches in length, and covering the flanged end with a small piece of muslin. An asbestos pulp is next prepared by boiling finely chipped asbestos with water, and an amount sufficient to cover the muslin is then sucked up on the closed end of the tube. In testing the supernatant liquid in the casserole, a very small portion is sucked through the asbestos filter, the tube being at once inverted in order to drop the contents on the porcelain surface. Operators should be careful to remove as little of the solution as possible at one time from the casserole, and to only commence testing the solution when it is apparent to the eye that the liquid is almost completely decolorized. Frequent tests and the use of large quantities of the solution in making tests tend to give results too high.

Instead of employing Wiley's tubes as explained above, a piece of tubing about the same dimensions is drawn out to a small bore at a point about an inch from end; a small plug of dry asbestos is now inserted in the short section of the tube next to the constriction, and the tube is ready for use in the manner before described.

In calculating results the following proportion can be used; number of cubic centimeters of solution required to precipitate the copper in 10 cubic centimeters Fehling's solution : amount in grams of glucose equivalent to 10 cubic centimeters, Fehling solution : : 1,000 : x. If 10 grams of the sample diluted to 100 cubic centimeters were used, x will give exact percentage of invert sugars in sample analyzed.

Determination of sucrose by inversion.—In analysis of sirups, molasses, etc., 5 grams of the samples are weighed up, washed into a flask, and diluted to 200 or 300 cubic centimeters; 5 cubic centimeters strong hydrochloric acid is then added, and the flask and contents are heated in a water bath for fifteen minutes at a temperature of 65° to 75° C. The solution is then neutralized with sodium carbonate, made up to a volume of one liter and invert sugar is determined as before described. The total invert sugar minus glucose previously determined, gives amount of glucose equivalent to sucrose present; this amount multiplied by .95, gives amount of sucrose contained, as determined by inversion. When it is desired to decolorize juices previous to determining glucose, or to determine glucose in a solution prepared for polarization, the excess of lead used in clarifying should be precipitated out by means of pulverized sodium carbonate, and the solution diluted to the requisite volume, and filtered before making the estimation.

The essential data to be given in stating the results of an analysis of juice are as follows:

Specific gravity.
Density, Degrees Brix or Beaume.
Total solids.
Sucrose.
Glucose.
Solids not sugars.
Co efficient of purity.
Glucose ratio.

The proportion of solids not sugars is ascertained by subtracting the sum of the per cents. of sucrose and glucose from per cent. of total solids. The co-efficient of purity is obtained by dividing the per cent. of sucrose by per cent. of total solids and multiplying by 100. The glucoose ratio, or ratio of glucose to sucrose, is obtained by dividing per cent. glucose by per cent. sucrose and multiplying by 100. The per cent. of available sugar[1] is ascertained by subtracting one and one-half times the per cent. of glucose from the per cent. of sucrose. "Pounds of available sugar per ton of cane" is obtained by multiplying 2,000 by the per cent. of available sugar, and the product by the per cent. of extraction. The per cent. of extraction is obtained by dividing weight of juice by weight of cane and multiplying by 100.

Re-agents—preparation of basic lead acetate.—Weigh up about 220 grams normal lead acetate and about 130 grams of litharge; boil with 700 to 800 cubic centimeters water for about one half hour, stirring well to promote solution and prevent bumping. Cool, dilute to 1 liter and siphon or filter off the clear supernatant liquid. Instead of the above proportions of lead acetate and oxide, 350 grams of lead subacetate (basic acetate), dissolved in water and diluted to 1,000 cubic centimeters may be employed.

Fehling's solution.—Weigh up accurately 34,640 grams of crytallized copper sulphate, C. P., dissolve in about 200 cubic centimeters water by the aid of a gentle heat and allow to cool. Also weigh up 180 grams crystallized sodium potassium tartrate (Rochelle salt) and 70 grams of caustic soda; dissolve in about 500 or 600 cubic centimeters water, heating gently; cool, and pour into it slowly the copper sulphate solution, stirring mixture well. Transfer to a liter flask and dilute to a bulk of 1,000 cubic centimeters. Preserve in a tightly stoppered bottle in a dark place. Solutions which allow the formation of a precipitate of copper suboxide on standing, or when diluted with water and boiled, should be rejected as untrustworthy.

Standard glucose solution.—The glucose solution used in standardizing Fehling's copper solution is best prepared by dissolving 2 grams of C. P. anhydrous glucose in distilled water, and diluting to a volume of 1 liter, thus giving a solution containing 2 milligrams of glucose per cubic centimeter.

In order to determine the true strength of the copper solution, 10 cubic centimeters are run into a casserole from a burette and are diluted to three or four times that bulk with water. The liquid is then heated to boiling, and the glucose solution run in gradually until the precipitation of the copper is complete, the end point of the reaction being determined as previously described under the head of glucose estimations. The number of cubic centimeters of the glucose solution taken, multiplied by 2, will give the number of milligrams of glucose required to precipitate the copper in 10 cubic centimeters of the Fehling's solution.

The potassium ferrocyanide solution is prepared by dissolving 80 grams in water and diluting to a bulk of 1 liter.

The dilute acetic acid used should be about 20 per cent. to 30 per cent. strength.

The dydrochloric acid used in inversion should have a specific gravity of 1.18–1.20.

Determination of ash.—Two to 3 grams of the sample is ignited at a red heat in a weighed platinum or nickel dish until destruction of organic matter is complete.

The sample, previous to icineration in a platinum dish, may be treated with about 1 cubic centimeter of concentrated sulphuric acid, the heat being applied gradually at first, and then to full redness. One-tenth must be deducted from the weight of the ash thus obtained, on account of the increase in weight caused by the combination of the sulphuric acid with some of the ash constituents.

Calculation of losses from inversion.—In calculating losses from inversion, the formula given by Dr. Stubbs, in the Louisiana Planter of March 30, 1889, is recommended, as by its use the amount of sucrose inverted can be estimated very closely, even where the weight of the juice only, as it comes from the mill, is known. The only data required in this determination is the weight of juice, and the per cents. of sucrose and glucose in the juice, and in the products of the different stages of the manufacture. The total amount of inversion, or the amount of sucrose inverted in any single operation, can be readily determined by the application of the formula as here given:

---

[1] NOTE.—This is purely empirical, and practical factory results have, in many instances, demonstrated that available sugar estimates are thus rendered too low.

Let s = per cent. sucrose in raw juice.
g = per cent. glucose in raw juice.
s' = per cent. of sucrose in clarified juice, sirup, or masse cuite.
g' = per cent. of glucose in clarified juice, sirup, or masse cuite.
w = weight of raw juice.

Let x = sucrose inverted between raw juice and clarified juice, sirup, or masse cuite, as the case may be.

Then $\frac{100}{95}x$ = glucose gained at same time, as 95 parts of sucrose on inversion yield 100 parts of glucose.

Then $ws - x$ = sucrose in clarified juice, sirup, or masse cuite.

And $wg + \frac{100}{95}x$ = glucose in same.

But in the clarified juice, sirup, or masse cuite, the total sucrose and glucose are to each other as s' is to g'; therefore,

$$ws - x : wg + \frac{100}{95}x :: s' : g'; \text{ from which } x = \frac{95\,w\,(sg' - s'g)}{95\,g' + 100}$$

This method of calculation can even be applied to the molasses by subtracting from ws, before substitution, the amount of *pure* sugar obtained by weighing and polarizing as it comes from the centrifugal.

Sampling.—As the chief object in taking samples is to obtain small fractional portions of the juice and the products of the various processes of the sugar-house, representing as nearly as possible in composition the whole, care should be observed to conduct the sampling in such a manner as regards time and quantity as to secure analytical results of as comprehensive a character as possible. In sampling juice small quantities should be taken from the trough at equal short intervals and thoroughly mixed, the mixture thus obtained being fairly representative of the juice extracted during the time occupied in taking samples In drawing samples of juice or sirup from tanks the contents should be well mixed by stirring before taking out the desired quantity for analysis. If several of these tanks, however, are to furnish a sample for a single analysis quantities proportional to the size of each tank should be taken and the whole well mixed. In sampling juice from clarifiers a mixture of equal quantities taken from several successive fillings of the clarifier will represent fairly well the whole bulk of juice sampled.

It is recommended that samples for analysis be taken as follows:

Raw juice.—A sample at least every six hours.

Sulphured juice.—Sample taken after heating; sampling before heating is considered unnecessary; tests should be made at same intervals as for raw juice.

Clarified juice.—Sample taken at same intervals as for raw juice.

Sirup.—Every tank should be sampled.

Sampling of masse cuite is to be condemned, as it has been found well nigh impossible to obtain samples homogeneous in character and representative of the whole. The true percentages of sucrose and glucose contained in the masse cuite can be closely determined by analyzing and weighing the sugar and molasses produced, and combining the results as follows:

Let w = weight of sugar.
w' = weight of molasses.
s = per cent. sucrose in the sugar.
g = per cent. glucose in the sugar.
s' = per cent. sucrose in molasses.
g' = per cent. glucose in molasses.

Then, by combining these terms in formulæ, we have the per cent. of sucrose in the masse cuite $= 100 \frac{(ws + w' s')}{w + w'}$ and per cent. of glucose $= \frac{100 (wg + w' g')}{w + w'}$

The control of diffusion work differs from that of mill work in that in addition to the sampling and analysis of the extracted juice the sugar content of both the fresh and exhausted cane must also be determined.

The fresh chips are passed through an ordinary hand-mill, and the analysis of the juice is conducted as usual. The per cent. of sucrose in the juice multiplied by the estimated average per cent. of juice in the cane and divided by 100 gives per cent. of sucrose in cane. The exhausted chips are also passed through the mill and the expressed juice tested. The per cent. of sucrose in exhausted chips subtracted from the per cent. sucrose in the fresh chips, divided by the latter per cent. and multiplied by 100 gives the per cent. of ratio of sucrose extracted to the total sucrose present.

Samples of juices and chips should be analyzed as expeditiously as possible after the sample is drawn, as, on standing, changes frequently take place which affect the value of the analytical results.

Determination of fiber.—Twenty grams of the fresh chips are weighed up and placed in a beaker or Erlenmeyer flask. The samples then washed repeatedly with water of a temperature of from 85° to 90° C., the washings each time being poured off. A temperature of the boiling point must be avoided in the early part of the operation, as coagulation of albuminoids will ensue and the extraction of soluble matters will thereby be hindered. The washings by decantation is continued until soluble albuminoids are dissolved out, when the contents of the flask are boiled with water several times, being treated with fresh portions after each decantation. The insoluble residue is now brought upon a weighed and dried muslin or asbestos filter, and the washing with boiling water is continued upon the filter. The filter and contents are then dried in air bath at 100° to 110° C., and weighed; the increase in weight is amount of fiber. Per cent. of fiber deducted from 100 gives per cent. of juice.

B. B. Ross.
Maurice Bird.
W. P. Martin.

METHODS OF ANALYSES OF DAIRY PRODUCTS.

BUTTER.

MICROSCOPIC EXAMINATION.

Place a small portion of the fresh sample taken from the inside of the mass on a slide, add a drop of pure sweet oil, cover with gentle pressure, and examine with a one-half to one-eighth inch objective for crystals of lard, etc.

Examine same specimen with polarized light and selenite plate without the use of oil.

Pure fresh butter will neither show crystals nor a parti-colored field with selenite. Other fats melted and cooled, and mixed with butter, will usually present crystals and variegated colors with the selenite plate.

For further microscopic study dissolve 4 or 5 cubic centimeters of the fat in 15 cubic centimeters of ether in a test tube. Stopper the tube with cotton wool loosely and allow to stand twelve to twenty-four hours at room temperature (20° to 25°). When crystals form at the bottom of the tube they are removed with a pipette or glass rod or tube, placed on a slide, covered and examined. The crystals formed by later deposits may be examined in a similar way.

SPECIFIC GRAVITY.

Weight of flask.—Use a small specific gravity flask of from 25 to 30 cubic centimeters capacity. The stopper should be beveled to a fine edge on top and the lower end should be slightly concave to avoid any trapping of air. The flask is to be thoroughly washed with hot water, alcohol, and ether, and then dried for thirty minutes to one hour at 100°. After cooling in a desiccator the weight of the flask and stopper is accurately determined.

Weight of water.—The flask in an appropriate holder (Fig. 1), conveniently made of galvanized iron, is filled with freshly boiled and still hot distilled water and placed in a bath of pure, very hot, distilled water in such a way that it is entirely surrounded by the liquid with the exception of the top.

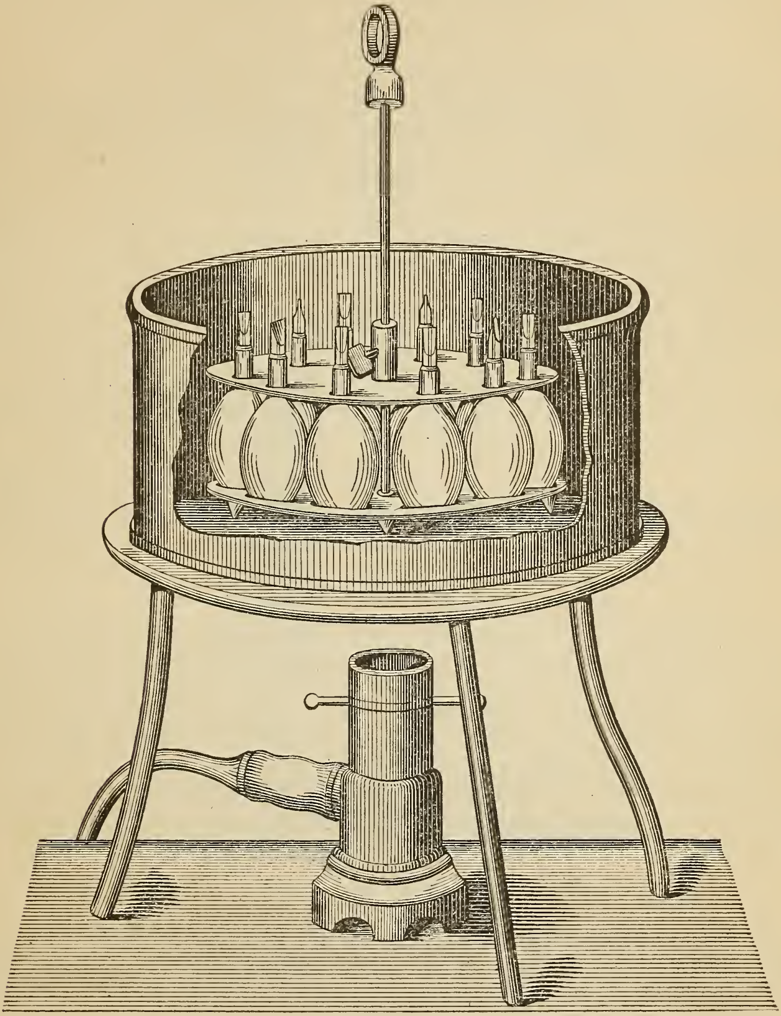

FIG. 1.

The water of the bath is kept in brisk ebullition for thirty minutes, any evaporation from the flask being replaced by the addition of boiling distilled water. The stopper[1] is then inserted, the flask removed, wiped dry, and after it is nearly cooled to room temperature, placed in the balance and weighed when balance temperature is reached. A convenient size of holder will enable the analyst to use eight flasks at once. The temperature at which water boils in each locality may also be determined; but unless at very high altitudes or on days of unusual barometric disturbance the variations will not be great, and will not appreciably affect the results.

---

[1] The stopper should be kept for a few minutes before use in hot distilled water.

Weight of fat at water boiling temperature.—The flask is emptied of its water, rinsed with alcohol and ether and dried again for a few minutes at 100°. It is then filled with the dry, hot, fresh filtered fat which should be entirely free from air bubbles.

The stoppered flask is then replaced in the water bath, kept for thirty minutes at the temperature of boiling water, removed and treated as above. The weight of fat having been determined, the specific gravity is obtained by dividing it by the weight of water previously found.

Example.

	Grams.
Weight of flask No. 22, dry	10.0197
Weight of flask No. 22, plus water	37.3412
Weight of water	27.3215
Weight of flask No. 22, plus fat	34.6111
Weight of fat	24.5914

Specific gravity=24.5914÷27.3215=.90008.

The weight of the flask dry and empty and the weight of water at 99° to 100° contained therein may be used constantly if great care be taken in handling and cleaning the apparatus.

Example.

	Grams.
Weight of flask No. 10, dry and empty	10.0028
Weight of flask after three weeks use	10.0030

MELTING POINT.

The apparatus for determining the melting point (shown in Fig. 2) consists of (1) an accurate thermometer for reading easily tenths of a degree; (2) a cathetometer for reading the thermometer (this may be done with an eyeglass, if held steadily and properly adjusted); (3) a thermometer; (4) a tall breaker glass, 35 centimeters high and 10 centimeters in diameter; (5) a test tube 30 centimeters high and 3.5 centimeters in diameter; (6) a stand for supporting the apparatus; (7) some method of stirring the water in the beaker, for example, a blowing bulb of rubber, and a bent glass tube extending to near the bottom of the beaker; (8) a mixture of alcohol and water of the same specific gravity as the fat to be examined.

MANIPULATION.

The disks of the fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube from a height of 15 to 20 centimeters on to a smooth piece of ice floating in water. The disks thus formed are from 1 to 1.5 centimeters in diameter and weigh about 200 milligrams. By pressing the ice under the water, the disks are made to float on the surface, whence they are easily removed with a steel spatula, which should be cooled in the ice water before using.

The mixture of alcohol and water is prepared by boiling distilled water and 95 per cent. alcohol for ten minutes to remove the gases which they may hold in solution. While still hot, the water is poured into the test tube already described until it is nearly half full. The test tube is then nearly filled with the hot alcohol. It should be poured in gently down the side of the inclined tube to avoid too much mixing. If the tube is not filled until the water has cooled, the mixture will contain so many air bubbles as to be unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top of the mixture.

The test tube containing the alcohol and water is placed in a tall beaker containing water and ice. The disk of fat is dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the alcohol-water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

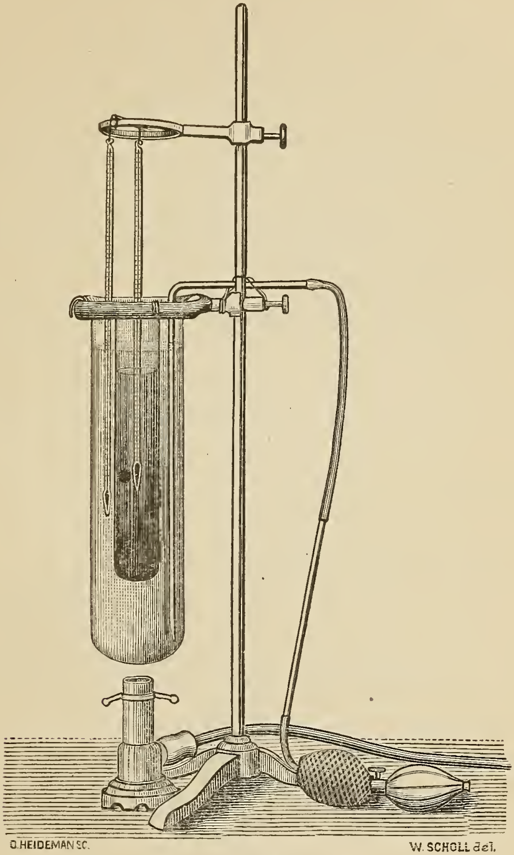

FIG. 2.

The delicate thermometer is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture in the vicinity of the disk the thermometer is moved from time to time in a circularly pendulous manner. A tube prepared in this way will be suitable for use for several days; in fact until the air-bubbles begin to attach themselves to the disk of fat. In no case did the two liquids become so thoroughly mixed as to lose the property of holding the disk at a fixed point, even when they were kept for several weeks.

In practice, owing to the absorption of air, it has been found necessary to prepare new solutions every third or fourth day.

The disk having been placed in position, the water in the beaker glass is slowly heated and kept constantly stirred by means of the blowing apparatus already described.

When the temperature of the alcohol-water mixture rises to about 6° below the melting point the disk of fat begins to shrivel and gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotary movement should be given to the thermometer bulb, which might be done with a kind of clock-work The rise of temperature should be so regulated that the last 2° of increment require about ten minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so the reading of the thermometer is to be made. As soon as the temperature is taken the test tube is removed from the bath and placed again in the cooler. A second tube, containing alcohol and water, is at once placed in the bath. The test tube (ice-water being used as a cooler) is of low enough temperature to cool the bath sufficiently. After the first determination, which should be only a trial, the temperature of the bath should be so regulated as to reach a maximum about 1°.5 above the melting point of the fat under examination.

Working thus with two tubes about three determinations can be made in an hour. After the test tube has been cooled the globule of fat is removed with a small cup attached to a wire before another disk of fat is put in.

The distilled water in which the piece of ice floats on which the disks are made should be recently boiled to free it of all air particles. It is important that the disks be free from air-bubbles.

The edge of the disk should not be allowed to touch the sides of the tube. This accident rarely happens, but in case it should take place and the disk adhere to the sides of the tube, a new trial should be made.

Triplicate determinations should be made and the second and third results should show a near agreement.

Example.

Melting point of sample of butter 6372 (No. 3):

	Co
(1)	33.15
(2)	33.05
(3)	33.00

ESTIMATION OF VOLATILE ACIDS.

RE-AGENTS.

SOLUTION OF CAUSTIC SODA.

1. 100 grams NaOH dissolved in 100 cubic centimeters pure water. The caustic soda should be as free as possible from carbonate and be preserved from contact with the air.
2. Alcohol, about 95 per cent. redistilled with caustic soda.
3. Solution of sulphuric acid containing 25 cubic centimeters strongest H_2SO_4 in 1,000 cubic centimeters of water.
4. An accurately standardized approximately decinormal solution of barium hydrate.
5. Alcoholic solution of phenolphthalein.

APPARATUS.

1. Saponification flasks of hard, well-annealed glass, capable of resisting the tension of alcohol vapor at 100°. The flask shown in Fig. 3 is most suitable for this purpose. It should have from 250 to 300 cubic centimeters capacity.

(*a.*) Instead of such a flask an Erlenmeyer flask of the same capacity fitted with long glass tube or reflux condenser may be used.

2. A pipette graduated to deliver 40 cubic centimeters.
3. Distilling apparatus as represented in Fig. 4. One with a smaller bulb tube may also be used.
4. An accurately calibrated burette reading to tenths of a cubic centimeter.

THE MANIPULATION.

Weighing the fat.—The butter or fat to be examined should be melted and kept in a dry warm place at about 60° for two or three hours until the moisture and curd have entirely settled out. The clean supernatant fat is poured off and filtered through a dry filter paper in a jacketed filter containing boiling water, to remove all foreign matter and any traces of moisture. Should the filtered fat in a fused state not be perfectly clear the treatment above mentioned must be repeated.

The saponification flasks are prepared by having them thoroughly washed with water, alcohol, and ether, wiped perfectly dry on the outside and heated for one hour to 100° (*circa*). The flasks should then be placed in a tray by the side of the balance and covered with a silk handkerchief until they are perfectly cool. They must not be wiped with a silk handkerchief within fifteen or twenty minutes of the time they are weighed. The weight of each flask is determined accurately, using a flask for a counterbalance or not, as may be convenient. The weight of the flasks having been accurately determined they are charged with the melted fat in the following way:

A pipette with a long stem marked to deliver 5.75 cubic centimeters is warmed to a temperature of about 50°. The fat having been poured back and forth once or twice into a dry beaker in order to thoroughly mix it, it is taken up in the pipette, the nozzle of the pipette carried to near the bottom of the flask, it having been previously wiped to remove any adhering fat. The 5.75 cubic centimeters of fat are then allowed to flow into the flask and the pipette is moved. After the flasks have been charged in this way they should be recovered with the silk handkerchief and allowed to stand fifteen or twenty minutes, when they are again weighed.[1]

The saponification.—Ten cubic centimeters of 95 per cent. alcohol redistilled from caustic soda are added to the fat in the flask, 2 cubic centimeters of the concentrated soda solution are added, a soft cork stopper inserted in the flask and tied down with a piece of twine. The saponification is then completed by placing the flasks upon the water or steam bath as indicated in Fig. 3. The flasks during the saponification, which should last for one hour, should be gently rotated from time to time, being careful not to project the soap for any distance up the sides of the flask. At the end of an hour the flasks, after having been cooled to near the room temperature, are opened.

Removal of the alcohol.—The stoppers having been laid loosely in the mouth of the flasks the alcohol, is removed by dipping the flasks into a steam bath. The steam should cover the whole of the flask except the neck. After the alcohol is nearly removed frothing may be noticed in the soap, and to avoid any loss from this cause or any creeping of the soap up the sides of the flask it should be removed from the bath and shaken to and fro until the frothing disappears. The last traces of alcohol vapor may be removed from the flask by waving it briskly, mouth down, to and fro. Complete removal of the alcohol with the precautions above noted should take about forty-five minutes.

Dissolving the soap.—After the removal of the alcohol the soap should be dissolved by adding 100 cubic centimeters of recently boiled distilled water, and warmed on the steam bath with occasional shaking until the soap is completely dissolved.

Setting free the fatty acids.—When the soap solution has cooled to about 60° or 70° the fatty acids are separated by adding 40 cubic centimeters of the dilute sulphuric acid solution mentioned above.

Melting the fatty acid emulsion.—The flasks should now be restoppered as in the first

---

[1] Before weighing the flasks any desiccating material used in the balance should be removed. If round bottom flasks are employed, a special form of holder must be used. This is made on the principle of a test-tube rack, the lower board of the tray being perforated so as to receive the round bottom of the flask and being protected by lugs so that on being placed on a table the bottom of the flasks do not touch the table.

Example:

	Grams.
Weight of counterbalanced flask No. 3	22 5904
Weight of counterbalanced flask No. 3+fat	27.6734
Weight of fat	5.0830

instance and the fatty acid emulsion melted by replacing the flasks on the steam bath. According to the nature of the fat examined the time required for the fusion of the fatty acid emulsions may vary from a few minutes to hours.

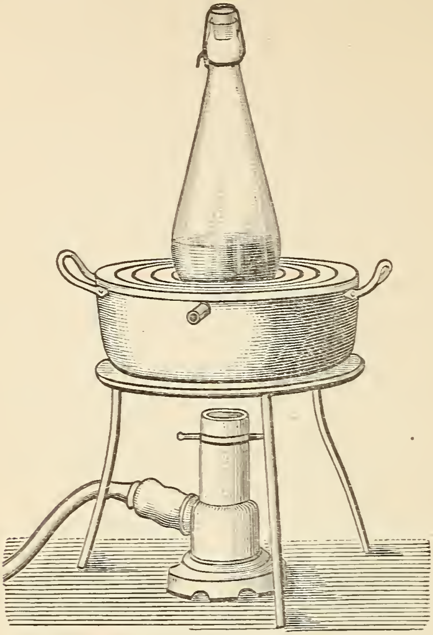

Fig. 3.

The distillation.—After the fatty acids are completely melted, which can be determined by their forming a transparent oily layer on the surface of the water, the flasks are cooled to room temperature and a few pieces of pumice stone added. The pumice stone is prepared by throwing it, at white heat, into distilled water and keeping it under water until used. The flask is now connected with a condenser (Fig. 4) slowly

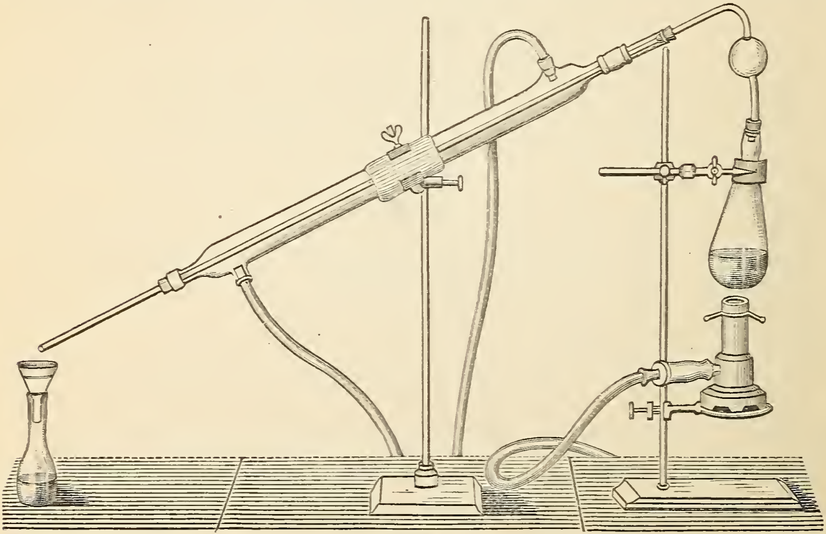

Fig. 4.

heated with a naked flame until ebullition begins, and then the distillation continued by regulating the flame in such a way as to collect 110 cubic centimeters of the distillate in as nearly as possible thirty minutes. The distillate should be received in flasks accurately graduated at 110 cubic centimeters.

Titration of the volatile acid.—The 110 cubic centimeters of distillate, after thorough mixing, are filtered through perfectly dry filter paper and collected in a flask graduated at 100 cubic centimeters. The 100 cubic centimeters of the filtered distillate are poured into a beaker holding from 200 to 250 cubic centimeters, .5 centimeters phenolphthalein solution added and decinormal barium hydrate run in until a red color is produced. The contents of the beaker are then returned to the measuring flask to remove any acid remaining therein, poured again into the beaker, and the titration continued until the red color produced remains apparently unchanged for two or three minutes.

ALTERNATE METHOD OF DETERMINING VOLATILE ACIDS.

SAPONIFICATION WITHOUT THE USE OF ALCOHOL.

To avoid the danger of loss from the formation of ethers and the trouble of removing the alcohol after saponification the fat may be saponified with a solution of caustic potash in a closed flask without alcohol. The operation is carried on exactly as indicated above for saponification in a closed flask, using caustic potash solution instead of soda, and omitting the operation for volatilizing the alcohol. The caustic potash is prepared as follows: Dissolve 100 grams of the purest potassium hydrate in 58 grams of distilled hot water. Allow to cool in a stoppered vessel, decant the clear caustic solution, and preserve in a vessel out of contact with the air. For the saponification use 2 cubic centimeters of the caustic potash solution, which is poured on the fat after it has solidified in the flask. Great care must be taken that none of the fat is allowed to rise on the sides of the saponifying flask to a point where it can not be reached by the alkali. During the process of saponification the flask can only be very gently rotated in order to avoid the difficulty mentioned above. This process is not recommended in any except a closed flask with round bottom. In the subsequent solution of the soap use only 80 cubic centimeters of distilled water and in setting free the fatty acids use 60 cubic centimeters of the dilute sulphuric acid. In other respects the distillation is conducted as described. Potash is used instead of soda, so as to form a softer soap and thus allow a more perfect saponification.

The saponification may also be conducted as follows: The alkali and fat in the melted state are shaken vigorously in the saponification flask until a complete emulsion is secured. The rest of the operation is then conducted as above.

IODINE EQUIVALENT.

RE-AGENTS.

1. Dissolve 25 grams of pure iodine in 500 cubic centimeters of 95 per cent. alcohol. Dissolve 30 grams of mercuric chloride in 500 cubic centimeters of 95 per cent. alcohol. The last solution, if necessary, is filtered and then the two solutions mixed. The mixed solution should be allowed to stand twelve hours before using.

2. *Decinormal hyposulphite of sodium solution.*—Take 24.6 grams of chemically pure hyposulphite of soda freshly pulverized as finely as possible and dried between filter or blotting paper. Make this up to 1,000 cubic centimeters at the temperature at which the titrations are to be made.

3. *Starch paste.*—1 gram of starch boiled in 200 cubic centimeters of distilled water for 10 minutes and cooled to room temperature.

4. *Solution of iodide of potassium.*—150 grams of iodide of potassium dissolved in water and made up to 1 liter.

5. *Solution of bichromate of potassium.*—Dissolve 3.874 grams chemically pure bichromate of potassium in distilled water and make the volume up to 1 liter at the temperature at which the titrations are to be made.

MANIPULATION.

Standardizing the hyposulphite of sodium solution.—Run 20 cubic centimeters of the bichromate of potassium solution into a glass stoppered flask to which has been added 10 cubic centimeters of the solution of iodide of potassium. Add to this 5 cubic centimeters of strong hydrochloric acid. Allow the solution of hyposulphite of sodium to flow slowly into the flask until the yellow color of the liquid has almost disappeared. Add a few drops of the starch paste and with constant shaking continue to add the hyposulphite of sodium solution until the blue color just disappears. The number of cubic centimeters of hyposulphite solution used multiplied by 5 is equivalent to 1 gram of iodine.

Example.—20 cubic centimeters $K_2Cr_2O_7$ solution required 16.2 cubic centimeters hyposulphite of soda: then $16.2\times5=81$ = number cubic centimeters of hyposulphite solution equivalent to 1 gram of iodine. Then 1 cubic centimeter hyposulphite solution = .0124 gram of iodine. Theory for decinormal solution of hyposulphite of sodium, 1 cubic centimeter = .0127 gram of iodine.

Weighing the sample.—About 1 gram of butter fat is to be weighed in a glass-stoppered flask holding about 300 cubic centimeters with the precautions mentioned for weighing the fat for determining volatile acids.

Absorption of iodine.—The fat in the flask is dissolved in 10 cubic centimeters of chloroform. After complete solution has taken place 30 cubic centimeters of the iodine-mercuric chloride solution are added. The flask is now placed in a dark place and allowed to stand with occasional shaking for three hours.

Titration of the unabsorbed iodine.—100 cubic centimeters of distilled water are added to the contents of the flask, together with 20 cubic centimeters of the iodide of potassium solution. Any iodine which may be noticed upon the stopper of the flask should be washed back into the flask with the iodide of potassium solution. The excess of iodine is now oxidized with the hyposulphite of sodium solution, which is run in gradually with constant shaking until the yellow color of the solution has almost disappeared. A few drops of starch paste are then added and the titration continued until the blue color has entirely disappeared. Toward the end of the reaction the flask should be stoppered and violently shaken so that any iodine remaining in solution in the chloroform may be taken up by the iodide of potassium solution in the water. A sufficient quantity of hyposulphite of sodium solution should be added to prevent a re-occurrence of any blue color in the flask for five minutes.

Setting the value of the iodine solution by the hyposulphite solution.—At the time of adding the iodine solution to the fats two blank flasks of the same size and nature as those used for the determination should be employed for conducting the operation described above without the presence of any fat. In every other respect the conduct of the blank experiments should be just as described. These blank experiments must be made each time the iodine solution is used.

Example.

BLANK DETERMINATIONS.

1. 30 cubic centimeters iodine solution requires 46.4 cubic centimeters hyposulphite of sodium solution.

2. 30 cubic centimeters iodine solution requires 46.8 cubic centimeters of hyposulphite of sodium solution.

Mean 46.6.

PERCENTAGE OF IODINE ABSORBED.

Weight of fat taken	grams	1.0479
Quantity of iodine solution used	cubic centimeters	30
Hyposulphite equivalent of iodine used	do	46.6
Hyposulphite to oxidize remaining iodine	do	14.7
Hyposulphite equivalent to iodine absorbed	do	31.9

Percentage of iodine absorbed, $31.9 \times .0124 \times 100 \div 1.0479 = 37.75$ per cent.

WATER.

Place about 2 grams in flat-bottomed platinum dish two-thirds full of clean, dry sand and heat for two hours in air bath at 105° C. (For alternate method of estimation of water, see page 81.)

ESTIMATION OF SALT.

Volumetric method.—The amount of the butter or butter substitute to be taken is from five to ten grams; weigh in a counterpoised beaker-glass. The butter (fresh from the refrigerator) is placed in portions of about 1 gram at a time in the beaker, these portions being taken from different parts of the sample. By this means a reasonably fair sample of the whole is obtained.

The given quantity having been weighed out, it is removed from the pan.

Hot water is now added (about 20 cubic centimeters) to the beaker containing the butter, and after it has melted the liquid is poured into the bulb of the separating apparatus. The stopper is now inserted and the contents shaken for a few moments.

After standing until the fat has all collected on top of the water, the stop-cock is opened and the water, containing most of the salt, is allowed to run into an Erlenmeyer flask, being careful to let none of the fat globules pass.

Hot water is again added to the beaker and thence poured into the separatory apparatus, the bottle well shaken, and the foregoing process is repeated from ten to fifteen times, using each time time 10 to 20 cubic centimeters of water.

The resulting washings contain all but a mere trace of the NaCl originally present in the butter.

Estimation of NaCl in filtrate.—The chloride of sodium is now determined in the filtrate by a standard solution of $AgNO_3$, using a few drops of a saturated solution of potassium chromate as indicator.

TOTAL MINERAL MATTER (ASH) AND CURD.

The methods of estimating curd depend on the principle of first drying a weighed portion of the butter and afterwards extracting the fat with ether or petroleum. The residual mass is then weighed and the curd determined by loss on ignition. This process is carried on as follows:

Five to ten grams of butter are dried at 100° C. for a few hours in a porcelain dish. The dried fat, etc., is filtered through a Gooch crucible, the contents of the dish all brought into the crucible and well washed with ether or light petroleum. The filter crucible is dried for two hours in air-bath and weighed. The curd is then determined by loss of weight on ignition.

ELECTIVE DETERMINATIONS.

For the further study of the properties of butter the following recommendations are made to be followed by the analyst or not as he may elect.

Reduction of silver.—To determine whether or not the butter has been derived from a cow fed on cotton-seed meal it will usually be sufficient to test the sample by Bechi's reagent.

The solutions used are as follows: Five grams of silver nitrate dissolved in 100 cubic centimeters, 95 per cent. alcohol, and 1 cubic centimeter of strong nitric acid added. The manipulation is conducted as follows: Measure out 5 cubic centimeters of the fat into a small porcelain dish and add 5 cubic centimeters of the above reagent. Stir together thoroughly with a stirring rod and place upon the steam bath for 10 minutes. Notice the changes of color which take place, the intensity of the color produced, and the amount of silver deposited as a mirror on the sides of the dish.

Reduction test applied to the fatty acids.—Instead of testing the original glycerides as above the test may be applied to the fatty acids in accordance with the following directions:

Reagent.—Dissolve 30 grams of nitrate of silver in 100 cubic centimeters of water.

Manipulation.—Place 5 cubic ccentimeters of the fatty acid in the porcelain dish which is used above and dissolve in 20 cubic centimeters of 95 per cent. alcohol. Add 2 cubic centimeters of the reagent, stir thoroughly together, and heat on the water bath as in the first instance.

Melting point of the fatty acids.—The melting point of the fatty acids may be determined as described in Part 4, Bulletin No. 13, page 448.

The melting point of the fatty acids can not be determined in the same apparatus and by the same methods as those described for the fats themselves, because the acids are soluble in alcohol. It should be remembered that the melting point of the fatty acids is slightly above that of the glycerides, and the first determination in every case should be solely for the purpose of determining approximately the temperature at which the fat melts.

The bulb of a delicate thermometer is coated with the fat acid, and the thermometer by means of a cork is fastened in a round flask of 250 cubic centimeters capacity. The bulb of the instrument should occupy as nearly as possible the center of the flask. The cork should have an air passage for the equalization of the pressure. The flask is slowly heated in a current of warm air, or otherwise, and as the melting point is approached a rotary motion is given to it. When the fat melts it is seen to collect in a small drop on the lowest part of the bulb, remaining stationary while the flask is turned. The thermometer is best held horizontally.

Crystallizing point of the fatty acids.—The crystallizing point of the fatty acid is determined by means of the method described in Bulletin No. 13, Part 4, page 447.

A very delicate thermometer with a long bulb is used. the thermometer being graduated into tenths of a degree ; the readings of the mercury are made with a small eye-glass. A test tube about 15 centimeters in length and 2.5 to 3 centimeters in diameter is filled with the melted fatty acids. The temperature at which the acid is melted should be sufficiently high to secure a complete liquefaction. The tube containing the fat is placed in a stopper carried in a bottle so that the whole of the fatty acid may be contained in that part of the tube protected from external currents of air by the bottle. The bottom of this protected bottle should be warm so that its temperature may be several degrees higher than the crystallizing point of the fatty acid. This precaution is necessary to avoid a too rapid crystallization of the fatty acid in the bottom of the test tube, and to secure as nearly as possible a uniform crystallization throughout the whole mass. The thermometer is suspended in such a manner that the bulb may occupy as nearly as possible the center of the fatty mass. The thermometer should be protected from currents of air and should be kept perfectly still. The position of the mercury in the thermometer is carefully watched by the attendant as it gradually sinks towards the crystallizing point. When the crystals of the acid begin to appear in the bottom and on the sides of the test tube the descent of the mercury will become very slow and finally cease. The

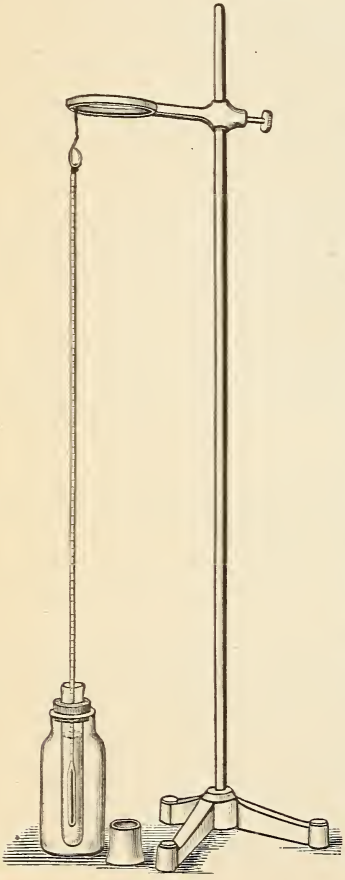

FIG. 5.

lowest point reached by the mercury should be noted. As the crystallization extends inward toward the bulb of the thermometer a point will be reached when the mercury will begin to rise. At that point, the test tube being held by the left hand, the thermometer should be taken by the right hand of the operator, and the partially crystallized mass of fat thoroughly stirred by turning the thermometer three or four times around the tube in both directions. Care should be exercised that at the end of this operation the bulb of the thermometer should hang as near as possible in the center of the crystallizing mass. Directly the above operation is accomplished the mercury will be seen to rise, and this rise of temperature will continue for some time, after which the mercury will remain stationary for one or two minutes. The highest point reached is taken as the true temperature of crystallization.

Refractive index.—The refractive index is conveniently determined by the apparatus of Professor Abbe, Fig. 6. The manipulation is conducted as follows: Fine tissue paper of rather heavy body is cut into rectangular pieces 3 centimeters in length by 1.5 centimeters in breadth. One of these pieces of paper is placed on the lower of the two glass prisms of the apparatus. Two or three drops of the oil or fat are placed upon the paper and the upper prism carefully placed in position so as not to move the paper from its place. In charging the apparatus with the oil in this way, it is placed in the horizontal position. After the paper disk holding the fat is secured by replacing the upper prism, the apparatus is placed in its normal position and the index moved until the light directed through the apparatus by the mirror shows the field of vision divided into dark and light portions. The dispersion apparatus is now turned until the rainbow colors on the part between the dark and light field have disappeared. Before doing this, however, the telescope, the eye-piece of the apparatus, is so adjusted as to bring the cross lines of the field of vision distinctly into focus. The index of the apparatus is now moved back and forth until the dark edge of the field of vision falls exactly in the intersection of the cross lines. The refractive index of the fat under examination is then read directly upon the scale by means of a small magnifying glass. To check the accuracy of the first reading, the dispersion apparatus should be turned through an angle of 180° until the colors have again disappeared and the scale of the instrument again read. These two readings should fall closely together, and their mean is the true reading of the fat under examination.

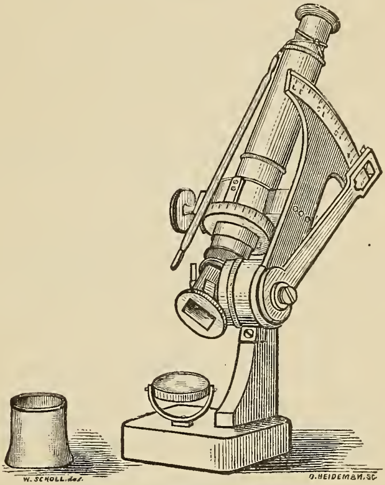
W. SCHOLL, del.
O. HEIDEMAN, SC.

FIG. 6.

For butter fats the apparatus should be kept in a warm place, the temperature of which does not fall below 30°. For reducing the results obtained to a standard temperature, say 25°, the following factor may be used. The mean rate of variation in the refractive index of butter fats for each degree of temperature was determined by me in twelve samples of butter fat (see Journal of Analytical Chemistry, Vol. 2, Part 3, July, 1888). This factor is .000176. As the temperature rises the refractive index falls.

Example.

Refractive index of a butter fat determined at 32° 4 =1.4540, reduced to 25°, as follows: 32.4—25=7.4=.0013; then 1.4540+.0013=1.4553.

The instrument used should be set with distilled water at 25°, the theoretical refractive index of water at that temperature being 1.3330. In the determination above given the refractive index of pure water measured 1.3300; hence the above numbers should be corrected for theory by the addition of 0.0030, making the corrected index of the butter fat mentioned at the temperature given, 1.4583.

Soluble and insoluble acids.—Use method described in Bulletin 13, Part 4, pages 455 to 461.

Saponification equivalent.—Use method described in Bulletin 13, Part 4, pages 461–462.

Rise of temperature with sulphuric acid.—Use method described in Bulletin 13, Part 4, pages 443–446.

METHODS FOR MILK ANALYSIS.

WATER.

Evaporate 1 to 2 grams of milk in a tarred flat platinum dish containing from 15 to 20 grams of pure dry sand, until apparently dry. Transfer to air bath and dry for one hour at temperature of boiling water. Cool in desiccator and weigh rapidly to avoid absorption of hygroscopic moisture.

TOTAL NITROGENEOUS MATTER.

Evaporate to dryness in the Kjeldahl digestion flask a known weight (5 grams) of milk and proceed exactly as described for this method by the nitrogen reporter.

FAT; METHOD OF ADAMS.

Coils made of thick filter paper, cut into strips 2.5 by 25 inches are thoroughly extracted with ether and alcohol. About 5 cubic centimeters of the well mixed milk are placed in a small beaker, covered with a watch glass, and weighed. The coil is then placed in the beaker until the contents are absorbed. The coil is removed and placed dry end down on a sheet of glass. The watch glass is replaced and the beaker again weighed, the loss in weight representing the milk absorbed by the coil. The coil on the piece of glass is dried at the temperature of boiling water for one hour, or better dried in hydrogen at temperature of boiling water to constant weight, transferred to a siphon extraction apparatus and extracted by at least 12 siphonings with absolute ether or petroleum spirit boiling at about 45°. The extracted fat is dried in hydrogen and weighed.

ALTERNATE METHOD OF ESTIMATING THE FAT IN MILK.

Method of Morse, Piggot, and Burton.—This method consists in the dehydration of the milk by means of anhydrous sulphate of copper; the extraction of the fat by means of the low-boiling products of petroleum; the saponification of the butter by means of an excess of a standard solution of potassium hydroxide in alcohol; and the determination of the excess of the alkali by means of a solution of hydrochloric acid. The following apparatus and re-agents are required:

(1) A porcelain mortar and pestle.

(2) An extraction tube, 14 or 15 millimeters in diameter, 220 millimeters in length, with funnel-shaped top. A straight chloride of calcium tube may be used for this.

(3) A 200-cubic centimeter Erlenmeyer flask, strong enough to be used with a filter pump.

(4) A suitable stand for holding the flask and extraction tube.

(5) Ten cubic centimeter pipettes.

(6) Weighing glasses with ground-glass stoppers.

(7) A low-boiling gasoline, distilling between 30° and 60° C.

(8) Dehydrated sulphate of copper.

(9) Seminormal solution of potash in 95 per cent. alcohol.

(10) A seminormal solution of hydrochloric acid.

Manipulation.—Place about 20 grams of the anhydrous copper sulphate, roughly measured in a copper spoon of the size to hold about that amount, in a porcelain mortar; make a cavity in the center of the mass with the pestle. Allow 10 cubic centimeters of the milk to run on to the copper sulphate, being careful that none of it touches the sides of the mortar. When the milk is nearly dry, grind the mass up with a little clean sand, transfer to the extraction tube, gently pressing it down in the tube by means of a glass rod. The lower portion of the extraction tube to be packed with clean cotton wool. The fat is extracted in the following way: 15 cubic centimeters of benzine are poured over the material in the extraction tube and drawn down, with the aid of the filter pump, until the whole of the mass to be extracted has become wet with the liquid, when the connection with the pump is closed; after about five minutes another portion of 15 cubic centimeters of benzine is poured into the tube and the whole of the liquid slowly drawn through with the aid of the pump into the flask. Usually one extraction of this kind is sufficient to withdraw the whole of the butter, but for the sake of greater accuracy the process may be repeated two or three times.

Titration.—The benzine may be evaporated and the residual butter fat saponified with about 25 cubic centimeters of the approximately seminormal potash. The residual alkali is determined by means of the seminormal hydrochloric acid, using phenolphthalein as indicator. The difference between the amount required in this process and the amount necessary to neutralize the quantity of alkali taken gives the amount of alkali required for the saponification. The number of milligrams of potash required for one gram of the fat is taken at 230. The fat may also be accurately titrated without evaporating the benzine.

ALTERNATE METHOD OF ESTIMATING WATER AND FAT IN MILK.

Method of Babcock.—In the bottom of a perforated test-tube is placed a clump of clean cotton, the tube is then filled three-quarters full of ignited asbestus, lightly packed, and a plug of cotton inserted over it. The tube and contents are weighed and the plug of cotton carefully removed and 5 grams of milk from a weighed pipette run into it, and the plug of cotton replaced. The tube connected at its lower end by a rubber tube and adapter with a filter pump is placed in a drying-oven of a 100° C., and a slow current of dry air drawn through it until the water is completely expelled, which in no case requires more than two hours.

The tube containing the solids from the above operation is placed in an extraction apparatus and exhausted with ether in the usual way.

ALTERNATE METHOD OF ESTIMATING WATER AND FAT.

Method of Professor Macfarlane.—A glass tube 4 to 5 cubic centimeters in length and 2 centimeters in diameter, open at one end, drawn out to a tube 5 millimeters in diameter at the other end, is two-thirds filled with asbestus fiber, such as is used in manufacturing packing. It is dried in the water bath for several hours, cooled in the desiccator, and weighed. Ten cubic centimeters of the milk is then added from a pipette, which is completely absorbed by the asbestus. It is then weighed, the additional weight of the milk representing the amount taken. The tube, along with many others, is placed in a water bath with constant level and dried for ten or twelve hours (during the night) at a temperature of 90°. Next morning the tubes are cooled in the desiccator and weighed, the loss in weight being the moisture. The tubes are then placed in the Soxhlet extraction apparatus and exhausted with petroleum ether for four hours. They are then removed and dried in a steam bath, cooled in desiccator, and weighed. The loss represents the butter fat.

THE ESTIMATION OF SUGAR.

The re-agents, apparatus, and manipulation necessary to give the most reliable results in milk-sugar estimation are as follows:

Re-agents.—(1) *Basic plumbic acetate*, specific gravity 1.97. Boil a saturated solution of sugar of lead with an excess of litharge, and make it of the strength indicated above. One cubic centimeter of this will precipitate the albumens in 50 to 60 cubic centimeters of milk.

(2) *Acid mercuric nitrate.* Dissolve mercury in double its weight of nitric acid, specific gravity 1.42. Add to the solution an equal volume of water. One cubic centimeter of this re-agent is sufficient for the quantity of milk mentioned above. Larger quantities can be used without affecting the results of polarization.

(3) *Mercuric iodide with acetic acid.* KI 33.2 grams, $HgCl_2$ 13.5 grams, H, C_2H_3O 20 cubic centimeters, H_2O 64 cubic centimeters.

Apparatus.—(1) Pipettes marked at 59.5, 60, and 60.5 cubic centimeters. (2) Sugar flasks marked at 102.4 cubic centimeters. (3) Filters, observation tubes, and polariscope. (4) Specific gravity spindle and cylinder. (5) Thermometers.

Manipulation.—(1) The room and milk should be kept at a constant temperature. It is not important that the temperature should be any given degree. The work can be carried on equally well at 15° C., 20° C., or 25° C. The slight variations in rotary power within the above limits will not affect the result for analytical purposes. The temperature selected should be the one which is most easily kept constant.

(2) The specific gravity of the milk is determined. For general work this is done by a delicate specific gravity spindle. Where greater accuracy is required, use specific gravity flask.

(3) If the specific gravity be 1.026, or nearly so, measure out 60.5 cubic centimeters into the sugar-flask. Add 1 cubic centimeter of mercuric nitrate solution, or 30 cubic centimeters mercuric iodide solution, and fill to 102.4 cubic centimeter mark. The precipated albumen occupies a volume of about 2.44 cubic centimeters. Hence the milk solution is really 100 cubic centimeters. If the specific gravity is 1.030, use 60 cubic centimeters of milk. If specific gravity is 1.034, use 59.5 cubic centimeters of milk.

(4) Fill up to mark in 102.4 cubic centimeter flask, shake well, filter, and polarize.

NOTES.—In the above method or analysis the specific rotatory power of milk-sugar is taken at 52.5, and the weight of it in 100 cubic centimeter solution to read 100 degrees in the cane sugar scale at 20.56 grams. This is for instruments requiring 16.19 grams sucrose to produce a rotation of 100 sugar degrees. It will be easy to calculate the number for milk-sugar, whatever instrument is employed.

Since the quantity of milk taken is three times 20.56 grams, the polariscopic readings divided by 3 give at once the percentage of milk sugar when a 200 millimeter tube is used.

If a 400 millimeter tube is employed, divide reading by 6; if a 500 millimeter tube is used, divide by 7.5.

Since it requires but little more time, it is advisable to make the analysis in duplicate and take four readings for each tube. By following this method gross errors of observation are detected and avoided.

By using a flask graduated at 102.4 for 60 cubic centimeters no correction for volume of precipitated caseine need be made. In no case is it necessary to heat the sample before polarizing.

ESTIMATION OF ASH.

Evaporate to dryness in a weighed platinum dish 20 cubic centimeters of milk from a weighing-bottle, to which 6 cubic centimeters of HNO_3 has been added, and burn in muffle at low red heat until ash is free from carbon.

METHODS OF WINE ANALYSIS, ETC.

I. Specific gravity.—This determination is made with a picnometer or a Westphal balance controlled by a picnometer. Temperature 25° C.

II. Alcohol.—One hundred cubic centimeters of wine at 25° C. distilled (preferably in glass) give + cubic centimeter alcohol.

III. Extract.—Fifty cubic centimeters of wine (at 25° C.) in case of sweet wines a less amount, are evaporated in a platinum dish on the water bath to the proper consistency and then dried in a drying oven at 100° C. to constant loss of weight. Constant loss of weight is assumed when three weighings with equal intervals between the first and second and second and third give equal differences between the successive weighings. Weighings are to be made at intervals of fifteen minutes.

[NOTE ON THE EXTRACT DETERMINATION.—So far as I am aware no satisfactory method has been given for the determination of the extract of wine. I think that the method of the Bavarian chemists is on the whole the best. As the object is for purpose of comparison with some standard, we can not do better I think than to follow the standard most generally adopted. I have in mind when the new chemical laboratory of the University of California is fitted up to make some experiments on the determination of this extract in a vacuum at a moderate temperature. I should modify somewhat the method of Gautier. However, until some better method has been established, I believe the method given will be most satisfactory.]

IV. *Acidity* (total acid constituents of the wine expressed as tartaric acid).—If carbonic acid is present, expel by shaking. Titrate with dilute alkali solution. The neutral point is determined by adding a drop of solution to delicate litmus paper.

V. *Volatile acids* (expressed as acetic acid).—Distil in a current of steam and titrate the carefully condensed distillate with standard alkali (decinormal).

VI. *Glycerine.*—(1) This is determined in dry wines as follows: The alcohol is driven off from 100 cubic centimeters wine, lime or magnesia added, and the same evaporated to dryness. The residue is boiled with 90 per cent. alcohol, filtered, and the filtrate evaporated to dryness. This residue is dissolved in 10 to 20 cubic centimeters alcohol, 15 to 30 cubic centimeters ether added, and the mixture allowed to stand until it is clear. It is then decanted from the sticky precipitate into a glass-stoppered weighing-bottle, evaporated to constant loss of weight and weighed.

(2) The following method is employed for sweet wines: One hundred cubic centimeters wine are measured into a porcelain dish and evaporated on the water bath to a sirupy consistence, mixed with 100 to 150 cubic centimeters absolute alcohol, poured into a flask, ether added in the proportion of 1½ volumes to each volume of alcohol used, the flask well shaken, and allowed to stand until the liquid becomes clear. This is then poured off and the residue again treated with a mixture of alcohol and ether. The liquids are mixed, the alcohol and ether driven off, the residue dissolved in water, and treated as in (1).

(3) In all glycerine determinations it is necessary to take into consideration the loss of glycerine due to its volatility with water and alcohol vapor, and accordingly to add to the glycerine found 0.100 gram for each 100 cubic centimeters of liquid evaporated.

(4) It is necessary to test the glycerine from sweet wines for sugar, and if any is

present it must be estimated by Soxhlet's or Knapp's method and its weight subtracted from that of the glycerine.

VII. *Sugar.*—This is to be determined by Sohxlet's or Knapp's method. The presence of unfermented cane sugar is to be shown by inversion, etc.

Polarization.—(1) The wine is decolorized with plumbic subacetate.

(2) A slight excess of sodic carbonate is added to the filtrate from (1). Two cubic centimeters of a solution of plumbic subacetate are added to 40 cubic centimeters white wine and 5 cubic centimeters to 40 cubic centimeters red wine, the solution is filtered and 1 cubic centimeter of a saturated solution of sodic carbonate added to 21 or 22.5 cubic centimeters of the filtrate.

(3) The kind of apparatus used and the length of the tube are to be given, and results estimated in equivalents of Wild's polaristrobometer with 200 millimeter tubes.

(4) All samples rotating more than .5 degrees to the right (in 220 millimeter tubes, after treating as above), and showing no change or but little change in their rotary power after inversion, are to be considered as containing unfermented glucose (starch sugar) residue.

(5) Rotary power of less than .3 degrees to the right shows that impure glucose has not been added.

(6) Wines rotating between .3 degrees and .5 degrees to the right must be treated by the alcohol method.

(7) Wines rotating strongly to the left must be fermented and their optical properties then examined.

VIII. *Tannin.*—Determine with Neubauer's permanganate method.

IX. *Potassic bitartrate.*—The determination of potassic bitartrate as such is to be omitted.

X. *Tartaric, malic, and succinic acids.*—(1) According to Schmidt and Hiepe's method.

(2) Determination of tartaric acid according to the modified Berthelot-Fleury method.

(3) If the addition of 1 gram finely-powdered tartaric acid to 100 grams wine produces no precipitate of potassic bitartrate, the modified Berthelot-Fleury method must be employed to determine free tartaric acid.

XI. *Coloring matter.*—(1) Only aniline dyes are to be looked for.

(2) Special attention is to be paid to the spectroscopic behavior of rosaniline dyes as obtained by shaking wines with amylic alcohol before and after saturation with ammonia.

XII. *Inorganic matter* (ash).—Burn in ordinary manner in flat platinum dish at as low a heat as possible; repeated moistening, drying, and heating to redness are advisable to get rid of all organic substances.

XIII. *Citric acid.*—Presence to be shown by a qualitative test, as baric citrate.

XIV. *Sulphuric acid.*—To be determined in the wine after adding hydrochloric acid.

XV. *Chlorine.*—To be determined in the nitric-acid solution of the burnt residue by Volhard's method.

XVI. *Lime, magnesia, and phosphoric acid.*—These are determined in the ash, fused with sodic hydrate and potassic nitrate, the phosphoric acid by the molybdenum method.

XVII. *Potash.*—Either in the wine ash, as the platinum double salt, or in the wine itself, by Kayser's method.

XVIII. *Gums.*—Presence shown by precipitation by alcohol; 4 cubic centimeters wine and 10 cubic centimeters 96 per cent. alcohol are mixed. If gum arabic has been added, a lumpy, thick, stringy precipitate is produced; whereas pure wine becomes at first opalescent and then floculent.

XIX. *Sulphurous acid.*—One hundred cubic centimeters of wine are distilled in a current of carbonic acid after the addition of phosphoric acid. The distillate carefully condensed is oxidized with bromine and the amount of H_2SO_4 determined.

ABNORMAL CONSTITUENTS OF WINES.

Mannite.—When present in any considerable quantity it can be detected by simply allowing the wine (a few drops will be sufficient) to evaporate spontaneously upon a glass slide or plate. The mannite will crystallize in a characteristic stellated form, which can be recognized at a glance. Should be allowed to stand twenty-four to forty-eight hours. When the quantity is small extract the residue evaporated nearly or quite to dryness with boiling 80 per cent. alcohol. Evaporate this extract to dryness and extract with 95 per cent. boiling alcohol, the mannite if present in any quantity will separate out in part upon cooling. If allowed to evaporate spontaneously the characteristic stellated crystals can be obtained.

Lactic acid.—This will be found in the alcohol ether extract of evaporated residue and can be recognized by usual tests. May be determined by titration with standard alkali. It may be necessary to take larger quantities of the wine for this investigation.

In diseased wines butyric acid may be tested for in the portion of volatile acid.

METHODS FOR THE DETERMINATION OF NITROGEN.

THE ABSOLUTE OR CUPRIC OXIDE METHOD.

[Applicable to all nitrogen determinations.]

The apparatus and re-agents needed are as follows:

APPARATUS.

Combustion tube of best hard Bohemian glass, about 26 inches long and one-half inch internal diameter.

Azotometer of at least 100 cubic centimeters capacity, accurately calibrated.

Sprengel mercury air-pump.

Small paper scoop, easily made from stiff writing-paper.

RE-AGENTS.

Cupric oxide (coarse).—Wire form; to be ignited and cooled before using.

Fine cupric oxide.—Prepared by pounding ordinary cupric oxide in mortar.

Metallic copper.—Granulated copper or fine copper gauze reduced and cooled in stream of hydrogen.

Sodium bicarbonate.—Free from organic matter.

Caustic potash solution.—Dissolve commercial stick potash in less than its weight of water so that crystals are deposited on cooling. When absorption of carbonic acid ceases to be prompt solution must be discarded.

LOADING TUBE.

Of ordinary commercial fertilizers take 1 to 2 grams for analysis. In the case of highly nitrogenic substances the amount to be taken must be regulated by the amount of nitrogen estimated to be present. Fill tube as follows: (1) About 2 inches of coarse cupric oxide. (2) Place on the small paper scoop enough of the fine cupric oxide to fill, after having been mixed with the substance to be analyzed, about 4 inches of the tube; pour on this the substance, rinsing watch glass with a little of the fine oxide, and mix thoroughly with spatula; pour into tube, rinsing the scoop with a little fine oxide. (3) About 12 inches of coarse cupric oxide. (4) About 3 inches of metallic copper. (5) About 2½ inches of coarse cupric oxide (anterior layer). (6) Small plug of asbestus. (7) Eight-tenths to 1 gram of sodium bicarbonate. (8) Large, loose plug of asbestus; place tube in furnace, leaving about 1 inch of it projecting; connect with pump by rubber stopper smeared with glycerine, taking care to make connection perfectly tight.

OPERATION.

Exhaust air from tube by means of pump. When a vacuum has been obtained allow flow of mercury to continue, light gas under that part of tube containing metallic copper, anterior layer of cupric oxide (see 5th above) and bicarbonate of soda. As soon as vacuum is destroyed and apparatus filled with carbonic acid gas, shut off the flow of mercury and at once introduce the delivery tube of the pump into the receiving arm of the azotometer, and just below the surface of the mercury seal of the azotomer, so that the escaping bubbles will pass into the air and not into the azotometer, thus avoiding the useless saturation of the caustic potash solution.

When the flow of carbonic acid has very nearly or completely ceased, pass the delivery tube down into the receiving arm so that the bubbles will escape into the azotometer. Light the jets under the 12-inch layer of oxide, heat gently for a few moments to drive out any moisture that may be present, and bring to red heat. Heat gradually mixture of substance and oxide, lighting one jet at a time. Avoid too rapid evolution of bubbles, which should be allowed to escape at rate of about one per second or a little faster.

When the jets under mixture have all been turned on, light jets under layer of oxide at end of tube. When evolution of gas has ceased turn out all the lights except those under the metallic copper and anterior layer of oxide, and allow to cool for a few moments. Exhaust with pump and remove azotometer before flow of mercury is stopped. Break connection of tube with pump, stop flow of mercury, and extinguish lights. Allow azotometer to stand for at least an hour or cool with stream of water until permanent volume and temperature are reached.

Adjust accurately the level of the KOH solution in bulb to that in azotometer, note volume of gas, temperature, and height of barometer; make calculations as usual. The labor of calculation may be much diminished by the use of the tables prepared by Messrs. Battle and Dancy, of the North Carolina Experiment Station (Raleigh, N. C.).

The above details are, with some modifications, those given in the report of the Connecticut Station for 1879 (p. 124), which may be consulted for details of apparatus, should such details be desired.

THE KJELDAHL METHOD.

[Not applicable in presence of nitrates.]

APPARATUS AND RE-AGENTS.

(1) Standard hydrochloric acid whose absolute strength has been determined: (*a*) by precipitating with silver nitrate and weighing the silver chloride as described in Crook's Select Methods, page 571; (*b*) by potassium tetroxalate which has been purified by recrystallizing two or three times; (*c*) by sodium carbonate, as described in Fresenius's Quantitative Analysis, second American edition, page 680; (*e*) by determining the amount neutralized by the distillate from a weighed quantity of pure ammonium chloride boiled with an excess of sodium hydrate. Half normal acid, *i. e.*, containing 18.23 grams hydrochloric acid to the liter, is recommended.

(2) Standard ammonia whose strength, relative to the acid, has been accurately determined. One-tenth normal ammonia, *i. e.*, containing .7 grams ammonia to the liter, is recommended for accurate work.

(3) "C. P." sulphuric acid, specific gravity 1.83, free from nitrates and also from

ammonia sulphate, which is sometimes added in the process of manufacture to destroy oxides of nitrogen. Eimer & Amend's "Strictly C. P." is good.

(4) Mercuric oxide, HgO, prepared in the wet way. That prepared from mercury nitrate can not safely be used.

(5) Potassium permanganate tolerably finely pulverized.

(6) Granulated zinc or pumice stone to be added to the contents of the flasks in distillation, when found necessary in order to keep from bumping.

(7) A solution of 40 grams of commercial potassium sulphide in one liter of water.

(8) A saturated solution of sodium hydrate free from nitrates, which are sometimes added in the process of manufacture to destroy organic matter and improve the color of the product. That of the Greenbank Alkali Company is of good quality.

(9) Solution of cochineal prepared according to Fresenius's Quantitative Analysis, second American edition, page 679.

(10) Kjeldahl digestion flasks of hard, moderately thick, well-annealed glass. These flasks are about 9 inches long, with a round, pear-shaped bottom, having a maximum diameter of 2½ inches, and tapering out gradually in a long neck, which is three-fourths of an inch in diameter at the narrowest part, and flared a little at the edge. The total capacity is 225 to 250 cubic centimeters.

(11) Distillation flasks of ordinary shape, 550 cubic centimeters capacity, and fitted with a rubber stopper and a bulb tube above to prevent the possibility of sodium hydrate being carried over mechanically during distillation. The bulbs are about 1½ inches in diameter, the tubes being the same diameter as the condenser and cut off obliquely at the lower end. This is adjusted to the tube of the condenser by a rubber tube.

(12) *A condenser.*—Several forms have been described, no one of which is equally convenient for all laboratories. The essential thing is that the tube which carries the steam to be condensed shall be of block-tin. The upper ends of the tin tubes should be bent so that the glass connections may have a slope toward the distilling flasks. All kinds of glass are decomposed by steam and ammonia vapor, and will give up alkali enough to impair accuracy. (See Kreussler and Henzold, Ber. Berichte, XVII, 34.) The condenser in use in the laboratory of the Connecticut Experiment Station, devised by Professor Johnson, consists of a copper tank, supported by a wooden frame, so that its bottom is 11 inches above the work-bench on which it stands. This tank is 16 inches high, 32 inches long, and 3 inches wide from front to back, widening above to 6 inches. It is provided with a water-supply tube, which goes to to the bottom, and a larger overflow pipe above. The block-tin condensing tubes, whose external diameter is three-eighths of an inch, seven in number, enter the tank through holes in the front side of it near the top, above the level of the overflow, and pass down perpendicularly through the tank and out through rubber stoppers tightly fitted into holes in the bottom. They project about 1½ inches below the bottom of the tank, and are connected by short rubber tubes with glass bulb tubes of the usual shape, which dip into precipitating beakers or Erlenmeyer flasks of about 300 cubic centimeters capacity. The titration can be made directly in them. The distillation flasks are supported on a sheet-iron shelf, attached to the wooden frame that supports the tanks in front of the latter. Where each flask is to stand a circular hole is cut, with three projecting lips, which support the wire gauze or asbestus under the flask, and three other lips, which hold the flask in place and prevent its moving laterally out of place while distillation is going on. Below this sheet-iron shelf is a metal tube carrying seven Bunsen burners, each with a stop-cock like those of a gas-combustion furnace. These burners are of a larger diameter at the top, which prevents smoking when covered with fine gauze to prevent the flame from striking back.

(13) The stand for holding the digestion flasks consists of a pan of sheet-iron 29 inches long by 8 inches wide, on the front of which is fastened a shelf of sheet-iron as long as the pan, 5 inches wide and 4 inches high. In this are cut six holes 1⅝ inches

in diameter. At the back of the pan is a stout wire running lengthwise of the stand, 8 inches high, with a bend or depression opposite each hole in the shelf. The digestion flask rests with its lower part over a hole in the shelf and its neck in one of the depressions in the wire frame, which holds it securely in position. Heat is supplied by low Bunsen burners below the shelf. With a little care the naked flame can be applied directly to the flask without danger.

THE DETERMINATION.

(1) *The digestion.*—Seven-tenths to 2.8 grams of the substance to be analyzed, according to its proportion of nitrogen, is brought into a digestion flask with approximately .7 gram of mercuric oxide and 20 cubic centimeters of sulphuric acid. The flask is placed on the frame above described in an inclined position, and heated below the boiling point of the acid for from 5 to 15 minutes, or until frothing has ceased. If the mixture froths badly, a small piece of paraffine may be added to prevent it. The heat is then raised until the acid boils briskly. No further attention is required till the contents of the flask have become a clear liquid, which is colorless, or at least has only a very pale straw color. The flask is then removed from the frame, held upright, and, while still hot, potassium permanganate is dropped in carefully and in small quantity at a time till, after shaking, the liquid remains of a green or purple color.

(2) *The distillation.*—After cooling, the contents of the flask are transferred to the distilling flask with about 200 cubic centimeters of water, and to this a few pieces of granulated zinc or pumice stone when found necessary to keep the contents of the flask from bumping, and 25 cubic centimeters of potassium sulphide solution are added, shaking the flask to mix its contents. Next add 50 cubic centimeters of the soda solution or sufficient to make the reaction strongly alkaline, pouring it down the side of the flask so that it does not mix at once with the acid solution. Connect the flask with the condenser, mix the contents by shaking, and distill until all ammonia has passed over into the standard acid. The first 150 cubic centimeters of the distillate will generally contain all of the ammonia. This operation usually requires from forty minutes to one hour and a half. The distillate is then titrated with standard ammonia.

The use of mercuric oxide in this operation greatly shortens the time necessary for digestion, which is rarely over an hour and a half in cases of substances most difficult to oxidize, and is more commonly less than an hour. In most cases the use of potassium permanganate is quite unnecessary, but it is believed that in exceptional cases it is required for complete oxidation, and in view of the uncertainty it is always used. Potassium sulphide removes all mercury from solution, and so prevents the formation of mercuro-ammonium compounds which are not completely decomposed by soda solution. The addition of zinc gives rise to an evolution of hydrogen and prevents violent bumping. Previous to use the re-agents should be tested by a blank experiment with sugar, which will partially reduce any nitrates that are present which might otherwise escape notice.

KJELDAHL METHOD MODIFIED TO INCLUDE THE NITROGEN OF NITRATES.[1]

[Applicable to all fertilizers containing nitrates.]

Besides the re-agents and apparatus given under the Kjeldahl method, there will be needed—

(1) Zinc dust. This should be an impalpable powder; granulated zinc or zinc filings will not answer.

(2) Commercial salicylic acid.

THE DETERMINATION.

Bring from .7 to 1.4 grams of the substance to be analyzed into a Kjeldahl digesting flask, add to this 30 cubic centimeters of sulphuric acid containing 2 grams of salicylic acid, and shake thoroughly; then add *gradually* 2 grams of zinc dust, shaking the contents of the flask at the same time. Finally place the flask on the stand for holding the digestion flasks, where it is heated over a low flame until all danger from frothing has passed. The heat is then raised until the acid boils briskly, and the boiling continued until white fumes no longer pour out of the flask. This requires about five or ten minutes. Add now approximately .7 gram mercuric oxide, and continue the boiling until the liquid in the flask is colorless, or nearly so. (In case the contents of the flask are likely to become solid before this point is reached, add 10 cubic centimeters more of sulphuric acid.) Complete the oxidation with a little permanganate of potash in the usual way, and proceed with the distillation as described in the Kjeldahl method. The re-ágents should be tested by blank experiments.

---

[1] Described by Prof. M. A. Scovell.

THE RUFFLE METHOD

APPARATUS AND RE-AGENTS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) A mixture of equal parts by weight of fine slaked-lime and finely powdered, crystallized sodium hyposulphite.

(3) A mixture of equal parts by weight of finely powdered granulated sugar and flowers of sulphur.

(4) Granulated soda-lime, as described under the soda-lime method.

(5) Combustion tubes of hard Bohemian glass, 20 inches long and ½ inch in diameter.

(6) Bulbed "U" tubes or Wills's bulbs, as described under the soda-lime method.

PREPARATION.

(1) Clean and fill the U tube with 10 cubic centimeters of standard acid.

(2) Fit cork and glass connecting tube. Fill the tube as follows: (1) A loosely fitting plug of asbestus, previously ignited, and then 1 to 1½ inches of the hyposulphite mixture. (2) The weighed portion of the substance to be analyzed is intimately mixed with from 5 to 10 grams of the sugar and sulphur mixture. (3) Pour on a piece of glazed paper, or porcelain mortar, a sufficient quantity of the hyposulphite mixture to fill about 10 inches of the tube; then add the substance to be analyzed, as previously prepared; mix carefully and pour into the tube; shake down the contents of the tube; rinse off the paper or mortar with a small quantity of the hyposulphite mixture and pour into the tube; then fill up with soda lime to within 2 inches of the end of the tube. (4) Place another plug of ignited asbestus at the end of the tube, and close with a cork. (5) Hold the tube in a horizontal position, and tap on the table until there is a gas channel all along the top of the tube. Make connection with the U tube containing the acid, aspirate, and see that the apparatus is tight.

The combustion.—Place the prepared combustion tube in the furnace, letting the open end project a little so as not to burn the cork. Commence by heating the soda-lime portion until it is brought to a full red heat. Then turn on slowly jet after jet toward the outer end of the tube, so that the bubbles come off two or three a second. When the whole tube is red hot and the evolution of the gas has ceased and the liquid in the U tube begins to recede towards the furnace, attach the aspirator to the other limb of the U tube, break off the end of the tube, and draw a current of air through for a few minutes. Detach the U tube and wash the contents into a beaker or porcelain basin, add a few drops of the cochineal solution, and titrate.

THE SODA-LIME METHOD.

[Not applicable in presence of nitrates.]

APPARATUS AND RE-AGENTS.

(1) Standard solutions and indicator the same as for the Kjeldahl method.

(2) Granulated soda-lime, fine enough to pass a 10-mesh sieve, and thoroughly dry.

(3) Fine soda-lime, fine enough to pass a 20-mesh sieve, also thoroughly dry.

To prepare soda-lime of the required fineness, the coarse granulated article of the trade may be ground until it will pass through a sieve of about ten hundredths inch mesh. It is then sifted on a sieve of about twenty hundredths-inch mesh to separate the fine from the coarse. The two portions are then thoroughly dried in the air-bath, or by heating in a porcelain dish or iron pan over a lamp, stirring constantly.

Excellent soda-lime may be easily and cheaply prepared according to the directions of Professor Atwater (Am. Chem. Journal, Vol. 9, p. 312), by slaking 2½ parts of quick-lime with a strong solution of 1 part commercial caustic soda (such soda as is used in the Kjeldahl process), care being taken that there is enough water in the solution to slake the lime. The mixture is then dried and heated in an iron pot to incipient fusion, and when cold, ground and sifted as above.

Instead of soda-lime Johnson's mixture of carbonate of soda and lime or slaked lime may be used.

Slaked lime may be granulated by mixing it with a little water to form a thick mass, which is dried in the water-oven until hard and brittle. It is then ground and sifted as above. Slaked lime is much easier to work with than soda-lime and gives excellent results, though it is probable that more of it should be used, in proportion to the substance to be analyzed, than is the case with soda-lime.

(4) Asbestus which has been ignited and kept in a glass-stoppered bottle.

(5) Combustion tubes about 40 centimeters long and about 12 millimeters internal diameter, drawn out to a point and closed at one end.

(6) Large bulbed "U" tubes with glass stop-cock, or Wills tubes with four bulbs.

THE DETERMINATION.

The substance to be analyzed should be powdered fine enough to pass through a sieve of 1 millimeter mesh; .7 to 1.4 grams, according to the amount of nitrogen present, is taken for the determination. Into the closed end of the combustion tube put a small, loose plug of asbestus, and upon it about 4 centimeters of fine soda-lime. In a porcelain dish or mortar mix the substance to be analyzed, thoroughly but quickly, with enough fine soda-lime to fill about 16 centimeters of the tube, or about 40 times as much soda-lime as substance, and put the mixture into the combustion tube as quickly as possible by means of a wide-necked funnel, rinsing out the dish and funnel with a little more fine soda-lime, which is to be put in on top of the mixture. Fill the rest of the tube to about 5 centimeters of the end with a granulated lime, making it as compact as possible by tapping the tube gently, while held in a nearly upright position during the filling. The layer of granulated soda-lime should be not less than 12 centimeters long. Lastly, put in a plug of asbestus about 2 centimeters long, pressed rather tightly, and wipe out the end of the tube to free it from adhering soda-lime.

Connect the tube by means of a well-fitting rubber stopper or cork with the "U" tube or Wills bulbs containing 10 cubic centimenters standard acid, and adjust it in the combustion furnace so that the end projects about 4 centimeters from the furnace, supporting the "U" tube or Willis tube suitably. Heat the portion of the tube containing the granulated soda-lime to moderate redness, and when this is attained extend the heat gradually through the portion containing the substance so as to keep up a moderate and regular flow of gases through the bulbs, maintaining the heat of the first part until the whole tube is heated uniformly to the same degree. Keep up the heat until gases have ceased bubbling through the acid in the bulbs and the mixture of substance and soda-lime has become white or nearly so, which shows that the combustion is finished. The combustion should occupy about three-quarters of an hour, or not more than one hour. Remove the heat, and when the tube has cooled below redness break off the closed tip and aspirate air slowly through the apparatus for two or three minutes to bring all the ammonia into the acid. Disconnect, wash the acid into a beaker or flask, and titrate with the standard alkali.

During the combustion, the end of the tube projecting from the furnace must be kept heated sufficiently to prevent the condensation of moisture, yet not enough to char the stopper. The heat may be regulated by a shield of tin slipped over the projecting end of the combustion tube.

It is found very advantageous to attach a Bunsen valve to the exit tube, allowing the evolved gases to pass out freely, but preventing a violent "sucking back" in case of a sudden condensation of steam in the bulbs.

METHODS OF DETERMINING POTASH.

METHOD OF LINDO AS MODIFIED BY GLADDING.

(1) *Superphosphates.*—Boil 10 grams of the fertilizer with 300 cubic centimeters of water for thirty minutes. Cool the solution; add a little oxalate of ammonia and then ammonia in slight excess, thus precipitating all phosphate and sulphate of lime, oxide of iron, and alumina, etc.; make up to 500 cubic centimeters, mix thoroughly and filter through a dry filter; take 50 cubic centimeters corresponding to 1 gram, evaporate nearly to dryness, add 1 cubic centimeter of dilute H_2SO_4 (1 to 1), and evaporate to dryness and ignite to whiteness. As all the potash is in form of sulphate, no loss need be apprehended by volatilization of potash, and a full red heat must be used until the residue is perfectly white. This residue is dissolved in hot water plus a few drops of HCl; 5 cubic centimeters of a solution of pure NaCl (containing 20 grams NaCl to the liter) and an excess of platinum solution (4 cubic centimeters) are now added. This solution is then evaporated to dryness in a small dish, the residue taken up with a little water, sufficient to dissolve it, and strong alcohol added. The precipitate is washed thoroughly with alcohol by decantation and on filter as usual. The washing should be continued even after the filtrate is colorless. Ten cubic centimeters of the NH_4Cl solution prepared as directed are now run through the filter, or the washing may be performed in the dish. These 10 cubic centimeters will contain the bulk of the impurities, and are thrown away. Fresh portions of 10 cubic centimeters NH_4Cl are now run through the filter several times (five or six). The filter is then washed thoroughly with pure alcohol, dried, and weighed as usual. The platinum solution used contains 1 gram metallic platinum in every 10 cubic centimeters.

(2) *Muriates of potash.*—In the analysis of these salts an aliquot portion, containing .500 gram, is evaporated with 10 cubic centimeters platinum solution plus a few drops of HCl, and washed as before.

(3) *Sulphate of potash, kainite, etc.*—In the analysis of these salts an aliquot portion containing .500 gram is taken, .250 gram of NaCl added, plus a few drops of HCl, and the whole evaporated with 15 cubic centimeters platinum solution. In this case special care must be taken in the washing with alcohol, to remove all the double chloride of platinum and sodium. The washing should be continued for some time after the filtrate is colorless. Twenty-five cubic centimeters of the NH_4Cl solution are employed instead of 10 cubic centimeters, and the 25 cubic centimeters poured through at least six times to remove all sulphates and chlorides. Wash finally with alcohol, dry, and weigh as usual.

To prepare the washing solution of NH_4Cl, place in a bottle 500 cubic centimeters H_2O, 100 grams of NH_4Cl; shake till dissolved. Now pulverize 5 or 10 grams of K_2PtCl_6, put in a bottle and shake at intervals for six or eight hours; let settle over night; then filter off liquid into a second bottle. The first bottle is then ready for a preparation of a fresh supply when needed.

ALTERNATE METHOD.

In case the potash is contained in organic compounds like tobacco-stems, cotton-seed hulls, etc., it is to be saturated with strong sulphuric acid and ignited in a muffle to destroy organic matter. Pulverize the fertilizer (200 or 300 grams) in a mortar;

take 10 grams, boil for ten minutes with 200 cubic centimeters water, and after cooling, and without filtering, make up to 1,000 cubic centimeters and filter through a dry paper. If the sample have 10 to 15 per cent. K_2O (kainite), take 50 cubic centimeters of the filtrate; if from 2 to 3 per cent. K_2O (ordinary potash fertilizers), take 100 cubic centimeters of the filtrate. In each case make the volume up to 150 cubic centimeters, heat to 100° and add, drop by drop, with constant stirring, slight excess of barium chloride; without filtering, in the same manner, add barium hydrate in slight excess. Heat, filter, and wash until precipitate is free of chlorides. Add to filtrate 1 cubic centimeter strong ammonium hydrate, and then a saturated solution of ammonium carbonate until excess of barium is precipitated. Heat. Add now, in fine powder, .5 gram pure oxalic acid or .75 gram ammonium oxalate. Filter, wash free of chlorides, evaporate filtrate to dryness in a platinum dish, and holding dish with crucible tongs, ignite carefully over the free flame below red heat until all volatile matter is driven off.

The residue is now digested with hot water, filtered through a small filter, and washed with successive small portions of water until the filtrate amounts to 30 cubic centimeters or more. To this filtrate, after adding two drops of strong hydrochloric acid, is added, in a porcelain dish, 5 to 10 cubic centimeters of a solution of 10 grams of platinic chloride in 100 cubic centimeters of water. The mixture is now evaporated on the water-bath to a thick sirup, or further, as above treated with strong alcohol, washed by decantation, collected in a Gooch crucible or other form of filter, washed with strong alcohol, afterwards with 5 cubic centimeters ether, dried for thirty minutes at 100° C., and weighed.

It is desirable, if there is an appearance of foreign matter in the double salt, that it should be washed, according to the previous method, with 10 cubic centimeters of the half-concentrated solution of NH_4Cl, which has been saturated by shaking with K_2PtCl_6, as recommended by Gladding.

The use of the factor .3056 for converting K_2PtCl_6 to KCl and .19308 for converting to K_2O are continued.

METHODS FOR DETERMINING PHOSPHORIC ACID AND MOISTURE.

(1) *Preparation of sample.*—The sample should be well intermixed and properly prepared and passed through a sieve having circular perforations one twenty-fifth of an inch in diamater, so that separate portions shall accurately represent the substance under examination, without loss or gain of moisture.

(2) *Determination of moisture.*—(*a*) In potash salts, nitrate of soda, and sulphate of ammonia heat 1 to 5 grams at 130° C. till the weight is constant, and reckon water from the loss. (*b*) In all other fertilizers heat 2 grams, or if the sample is too coarse to secure uniform lots of 2 grams each, 5 grams, for five hours at 100° in a steam bath.

(3) *Water-soluble phosphoric acid.*—Weigh out 2 grams in a small beaker, wash by decantation four or five times with not more than from 10 to 15 cubic centimeters of water, then rub it up in the beaker with a rubber-tipped pestle to a homogeneous paste, and then wash four or five times by decantation with from 10 to 15 cubic centimeters of water. Transfer the residue to a 9 cubic centimeter, No. 589 Schleicher and Schüll filter, and wash with water until the filtrate measures not less than 250 cubic centimeters. Mix the washings. Take an aliquot (corresponding to ½ gram) and determine phosphoric acid, as under total phosphoric acid.

(4) *Citrate-insoluble phosphoric acid.*—Wash the residue of the treatment with water into a 200 cubic centimeter flask with 100 cubic centimeters of strictly neutral ammonium citrate solution of 1.09 density, prepared as hereafter directed. Cork the flask securely and place it in a water bath, the water of which stands at 65° C. (The water bath should be of such a size that the introduction of the cold flask or flasks shall not cause a reduction of the temperature of the bath of more than 2° C.) Raising the temperature as rapidly as practicable to 65° C., which is subsequently maintained,

digest for thirty minutes from the instant of insertion, shaking every five minutes, filter the warm solution quickly (best with filter-pump), and wash with water of ordinary temperature. Transfer the filter and its contents to a capsule, ignite until the organic matter is destroyed, treat with 10 to 15 cubic centimeters of concentrated hydrochloric or nitric acid, digest over a low flame until the phosphate is dissolved, dilute to 200 cubic centimeters, mix, pass through a dry filter, take an aliquot and determine phosphoric acid as under total.

In case a determination of citrate-insoluble phosphoric acid is required in non-acidulated goods, it is to be made by treating 2 grams of the phosphatic material, without previous washing with water, precisely in the way above described, except that in case the substance contains much animal matter (bone, fish, etc.), the residue insoluble in ammonium citrate is to be treated by one of the processes described below.

(5) *Total phosphoric acid.*—Weigh 2 grams and treat by one of the following methods: (1) Evaporation with 5 cubic centimeters magnesium nitrate, ignition, and solution in acid. (2) Solution in 30 cubic centimeters concentrated nitric acid with a small quantity of hydrochloric acid. (3) Add 30 cubic centimeters concentrated hydrochloric acid, heat, and add cautiously and in small quantities at a time about .5 gram of finely pulverized potassium chlorate.

Boil gently until all phosphates are dissolved and all organic matter destroyed; dilute to 200 cubic centimeters; mix and pass through a dry filter; take 50 cubic centimeters of filtrate; neutralize with ammonia (in case hydrochloric acid has been used as a solvent add about 15 grams dry ammonium nitrate or its equivalent). To the hot solutions for every decigram of P_2O_5 that is present, add 50 cubic centimeters of molybdic solution. Digest at about 65° C. for one hour, filter, and wash with water or ammonium nitrate solution. (Test the filtrate by renewed digestion and addition of more molybdic solution.) Dissolve the precipitate on the filter with ammonia and hot water and wash into a beaker to a bulk of not more than 100 cubic centimeters. Nearly neutralize with hydrochloric acid, cool, and add magnesia mixture from a burette; add slowly (one drop per second), stirring vigorously. After fifteen minutes add 30 cubic centimeters of ammonia solution of density .95. Let stand several hours (two hours is usually enough). Filter, wash with dilute ammonia, ignite intensely for ten minutes, and weigh.

(6) *Citrate-soluble phosphoric acid.*—The sum of the water-soluble and citric insoluble subtracted from the total gives the citrate-soluble.

PREPARATION OF RE-AGENTS.

(1) *To prepare ammonium citrate solution.*—Mix 370 grams of commercial citric acid with 1,500 cubic centimeters of water; nearly neutralize with crushed commercial carbonate of ammonia; heat to expel the carbonic acid; cool; add ammonia until exactly neutral (testing by saturated alcoholic solution of coralline) and bring to volume of two liters. Test the gravity, which should be 1.09 at 20° C., before using.

(2) *To prepare molybdic solution.*—Dissolve 100 grams of molybdic acid in 400 grams or 417 cubic centimeters of ammonia of specific gravity .06, and pour the solution thus obtained into 1,500 grams or 1,250 cubic centimeters of nitric acid of specific gravity 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° C. deposits no yellow precipitate of ammonium phospho-molybdate. Decant the solution from any sediment, and preserve in glass stoppered vessels.

(3) *To prepare ammonium nitrate solution.*—Dissolve 200 grams of commercial ammonium nitrate in water and bring to a volume of 2 liters.

(4) *To prepare magnesia mixture.*—Dissolve 22 grams of recently ignited calcined magnesia in dilute hydrochloric acid, avoiding excess of the latter. Add a little calcined magnesia in excess, and boil a few minutes to precipitate iron, alumina and phos-

phoric acid; filter: add 280 grams of ammonium chloride, 700 cubic centimeters of ammonia of specific gravity .96, and water enough to make the volume of 2 liters. Instead of the solution of 22 grams of calcined magnesia 110 grams of crystallized magnesium chloride ($MgCl_2, 6H_2O$) may be used.

(5) *Dilute ammonia for washing.*—One volume ammonia of specific gravity .96 mixed with three volumes of water, or usually one volume of concentrated ammonia with six volumes of water.

(6) *Nitrate of magnesia.*—Dissolve 320 grams of calcined magnesia in nitric acid, avoiding an excess of the latter; then add a little calcined magnesia in excess; boil; filter from excess of magnesia, ferric oxide, etc., and bring to volume of 2 liters.

METHOD OF ANALYSIS OF CATTLE FOODS.

PREPARATION OF THE SAMPLE.

The substance is to be ground till all of it will pass through a sieve with 36 meshes to the linear inch, or 14 meshes to the centimeter.

MOISTURE.

Dry 2 to 3 grams of the substance heated fully to the temperature of boiling water, to constant weight in a current of dry hydrogen, but without allowing the glass containing the substance to come in contact with the boiling water.

PURE ASH.

Char the substance at a low red heat, which is conveniently done in a Fletcher gas muffle furnace heated, not by the large gas burner that is supplied with this furnace, but by a Fletcher solid flame burner (No. 46*b*); exhaust this charred mass with water; collect the insoluble residue on a filter; burn it; add this ash to the residue from the aqueous extract of the char; dry the whole at 110° and weigh. Determine carbon dioxide in this residue by the method of absorbing the thoroughly dried gas, expelled by sulphuric acid, in a weighed potash solution; the efficiency of the drying apparatus should be fully assured. Subtract the carbon dioxide found from the total residue.

ETHER EXTRACT.

Dry 2 grams of the substance four hours by heating fully to the temperature of boiling water, in a current of dry hydrogen. Exhaust with anhydrous and alcohol free ether until extraction is found by test to be complete. Dry the extract by exposure to the full heat of boiling water, in a current of dry hydrogen to constant weight.

CRUDE PROTEIN.

Determine nitrogen by the Kjeldahl method exactly in the manner described by the nitrogen reporter, and multiply the result by 6.25 for the crude protein. The standard of the alkalimetric solution used is to be verified by titration against the potassium tetroxalate sent with the samples, and the temperature of the solution should be approximately the same when used in the analysis as when standardized.

ALBUMINOID NITROGEN.

Stutzer's method. Prepare cupric hydrate as follows: Dissolve 100 grams of pure cupric sulphate in 5 liters of water, and add 2.5 cubic centimeters of glycerine; add dilute solution of sodium hydrate until the liquid is alkaline; filter; rub the precipitate up with water containing 5 cubic centimeters of glycerine per liter, and then wash by decantation or filtration until the washings are no longer alkaline. Rub the precipitate up again in a mortar with water containing 10 per cent. of glycerine, thus preparing a uniform gelatinous mass that can be measured out with a pipette. Determine the quantity of cupric hydrate per cubic centimeter of this mixture. To 1 gram of the substance add 100 cubic centimeters of water in a beaker, heat to boiling, or in the case of substances rich in starch heat on the water-bath ten minutes, add a quantity of cupric hydrate mixture containing .7 to .8 grams of the hydrate, stir thoroughly, filter when cold, wash with cold water, and without drying put the filter and its contents into the concentrated sulphuric acid for the determination of nitrogen after Kjeldahl. For the above filtration use Schleicher and Schüll's No. 589 paper, or Swedish paper, either of which contains so little nitrogen that it can be left out of account.

If the substance examined consists of seed of any kind, or residues of seeds, such as oil-cake or anything else rich in alkaline phosphate, add a few cubic centimeters of a concentrated solution of alum just before adding the cupric hydrate and mix well by stirring. This serves to decompose the alkaline phosphate. If this is not done cupric phosphate and free alkali may be formed and the protein copper may be partially dissolved in the alkaline liquid.

CRUDE FIBER.

Extract 2 grams of the substance with ordinary ether, at least nearly completely. To this fat-free residue, in a 500 cubic centimeter Erlenmeyer flask, add 200 cubic centimeters of boiling 1.25 per cent. sulphuric acid; connect the flask with a return flow condenser, and also with the air-blast, both tubes passing only a short distance beyond the rubber stopper into the flask. Boil at once, and continue the boiling for thirty minutes. The blast of air serves to reduce the frothing of the liquid, and the volume of the liquid is kept constant by means of the condenser.

While this boiling is going on, a filter may be prepared in one of the following ways:

(1) Pack firmly in the throat of a 90-millimeter funnel a layer of glass wool, 1 centimeter thick, by means of a glass rod, while water is poured in; over this make a thick welt of coarse asbestus pulp with the aid of strong suction.

(2) In the throat of a 90-millimeter funnel put a perforated disk of heavy platinum foil, about 2 centimeters in diameter; cover this first with a thin layer of glass wool, and above this make a thick welt of coarse asbestus pulp by strong suction. The first filter answers perfectly well for coarse fodders; the other is more suitable for oil meals and milled products generally. Suction is not to be used with either filter. The washing should be done, especially in the case of the concentrated fodders, almost wholly by decantation, and the final filtration should be made with as small a quantity of liquid as possible. If this is not done, the liquid should be well agitated just before pouring it on the filter so as to mix the coarser and finer portions of the substance. Clogging of the filter by fine particles is then less liable to take place. A turbidity which is sometimes observed in the acid filtrate from concentrated fodders has always been found in this laboratory to disappear when the liquid is made alkaline, and probably it has no effect on the final result. (W. P. Cutter.)

Collect the substance on one or the other of these filters, wash it thoroughly with boiling water, until the washings are no longer acid, rinse it back into the flask again with 200 cubic centimeters of a boiling 1.25 per cent. solution of sodium hydrate free from carbonate or almost entirely so, raise at once to boiling, boil thirty minutes in the manner described above, filter through a Gooch crucible, and wash with boiling water until the washings are neutral; wash then with alcohol, finally with ether, dry at 110°, weigh, incinerate perfectly, and give the loss of weight for crude fiber.

The carbonate-free alkali for this operation can be obtained of the Troy Laundry Machinery Company, 32 Dey street, New York, or 297 Wabash avenue, Chicago, under the name of Greenbank Alkali Company, double refined, 98 per cent. caustic soda, it being the same as that recommended for the Kjeldahl nitrogen method.

The acid and alkaline solutions are to be made up of the strength given accurately by titration, and not merely by the areometer.

On account of the great hygroscopicity of many vegetable substances, at the same time that the portions are weighed out for the determination of the moisture all the other portions required for the analysis should be weighed out also.

With reference to the advisability of substituting the much less expensive petroleum ether for anhydrous sulphuric ether in the extraction of the fat, all the chemists are requested to make determinations of the fat in the samples sent out with this solvent also, in the same manner as with the sulphuric ether. For this purpose procure the 25°–45° petroleum ether, and refractionate it, using only that portion which comes over at 40° to 45°. It will be best to weight the substance down in the extractor, in this operation.

INDEX.

A.

Page.

- Absolute ether, method of preparation of 35
  - results obtained by extracting with 36
- Acidity of wines, determination of 215
- Acids, fatty volatile, of butter, contribution to the study of 104
  - strength of, to be used 42
  - volatile, quantity of, in butter fat 99
    - variations of, in butter fat 111
- Albuminoids, estimation of, in milk 169
- Alcohol, determination of, in wines 215
  - withdrawal of, from bond for scientific purposes 71–75
- Alkali, purity of 43
  - strength of, to be used 42
- Ammonia, dilute, for phosphoric acid determinations 226
- Ammonium citrate, remarks on the use of 65
    - solution, preparation of 225
  - nitrate solution, preparation of 225
- Analyses of cane juice, essential data to be given in stating 197
- Analysis, official methods of 193
- Anderson, [illegible] J. T., letter from, in regard to butter analyses 127, 128
- A[illegible]ner, use of 40
- Arkansas experiment station, notes from, on butter analyses 134–136
- Ash, determination of, in cattle foods 16
  - sugar analysis in 198
  - estimation of, in milk 215
  - in cattle foods, remarks on 26, 27
- Atwater, Mr. W. O., discussion of remarks of, in regard to chemical study of foods and feeding stuffs 75–79
  - remarks of, on the lines of investigations for chemists 69–71
- Available sugar, estimation of 198

B.

- Babcock, Mr. S. M., letter from, in regard to butter analyses 119–121
- Banquet to members of the association 140
- Boiling point of water, drying at the 39–42
- Bowman, Mr. Walker, letter from, in regard to butter analyses 128–131
- Burney, Mr. W. B., letter from, in regard to butter analyses 128
  - remarks of, on the cause of breakage of distillation flasks (Kjeldahl method) 97
- Butter analysis 100
  - elective determinations 207
  - estimation of salt 207

Page.

- Butter analysis, iodine equivalent 207
    - reduction of silver 207
    - total mineral matter 207
  - examination of, Dronot's method 109
  - from cotton-seed, compared with that of other feed 143
  - influenee of food, breed, idiosyncrasy on the composition of 142
  - Mansfeld's method for the examination of 138
  - melting point of 202
  - method for the analysis of 99
  - microscopic examination of 200
  - natural, distinction of, from artificial 107
  - results of the examination of by Mansfeld's method 138
  - specific gravity of 200
- Butter fat analysis, Wollny's method of 101
  - and substances used in its adulteration, results of examinations of samples of 113
  - instructions to members of the association in regard to examination of samples of 113
  - results of examinations of samples by the Division of Chemistry 113–117
- Butters, abnormal, from Ireland, Denmark, and Sweden 110
    - collected details of so-called 111
  - discussion of methods for the examination of 138–140
  - Dubernard's method for the examination of 103
  - mixed, application of the methods of Reichert, Meissl, and Wollny to 105
- Butyrometer, a new 157

C.

- Caldwell, Mr. G. C., report of 15
- Carbonate, presence of, in alkali 43
- Carbonic acid in ash, determination of 39
- Casein in milk, estimation of, by Kjeldahl method 170
- Cattle foods, analysis of, in 1888–'89, report on 22–26
  - determination of crude protein in 226
    - ether extract of 226
    - pure ash in 226
  - discussion of report on 29
  - drying of, in a current of air 31
    - carbonic acid 31–34
    - hydrogen 30–34
    - over sulphuric acid 31
  - methods for the analysis of 226
  - moisture in 226
  - preparation of the sample of 226
  - report on analysis of 15
- Cereals, incorporation of, methods of analysis of, with cattle foods 33
- Cheese and ice-cream 156
- Chemical society, report of the committee on the organization of a national 65
- Chlorate of potash, use of, in destroying organic matter 50
- Chlorophyl, extraction of 36
  - food value of 40
- Citrate of ammonium, discussion of method of preparation of 61, 62
  - remarks on the use of 65
- Citric acid in wine 216
- Clarke, Mr. F. W., remarks of on the organization of a national chemical society 66–68
- Clerget's constant, revised figure of 195

Page.
Condensed milk, composition of 149
Constitution of the Association of Official Agricultural Chemists 191
Cotton-seed meal, feeding experiments to determine the influence of, on butter. 145, 146
Crude fiber 227, 228
Crystallizing point of fatty acids 210
Cutter, Mr. W. P., letter from, in regard to butter analyses 119

D.

Dairy products, methods of analysis of 200
Data, comparison of, obtained in the analyses of butters 136–138
Diphenylamine reaction, in the examination of milk, the significance of 168, 169

E.

Ether extract, in cattle foods 16, 17
remarks on 27
meaning of 33, 34
Extract, determination of, in wines 215

F.

Fat, Adams's method for the estimation of, in milk 212
alternate methods for the estimation of, in milk 212, 213
estimation of, in curdled milk 162
relative amount of, in first and last milkings 150
Fats, results obtained by different analysts in the examination of samples of .. 137
Fatty acids, crystallizing point of 210
Fehling solution 198
Fiber, determinations of, in cattle foods 18, 19
in cattle foods, remarks on 28–32
in sugar, analysis 200
Finney, Mr. B. F., letter from, in regard to butter analysis 133, 134
Food, influence of, on the composition of butter 140, 142
Foods and feeding stuffs, appointment of a committee to provide for a more thorough study of 94
influence of different, upon the melting point and chemical composition of butter 141
Formulæ for calculating the quantity of butter in mixtures 106, 107
Frear, Mr. Wm., letter from, in regard to butter analyses 133

G.

Gaines, Mr. Richard H., recommendations of, as reporter on phosphoric acid .. 50
report of, on phosphoric acid 44–49
Glucose, determination of 195, 197
solution, standard 198
Glycerine, determination of, in wines 215

H.

Harrington, Mr. H. H., letter from, in regard to butter analyses 131
Herff, Mr. B. von, letter from, in regard to butter analyses 121
Hydrogen, methods of using, for drying 38

I.

Ice-cream and cheese 156
Inversion, calculation of losses from 198

J.

Page.

- Jenkins, Mr. E. H., letter from, in regard to butter analyses 118
  - method of saponification of, adopted as an alternate method with the Mansfeld process 140
  - recommendations by, in regard to Mansfeld process 140
  - report of, on potash 79–83

K.

- Kedzie, Mr. R. C., letter from, in regard to butter analyses 124–127
- Kjeldahl method for nitrogen 85, 218, 219
  - modified to include nitrogen of nitrates 220, 221

L.

- Lactometer, the, its utility and reliability 156
- Ladd, Mr. E. F., letter from, in regard to butter analyses 119
- Lead acetate, basic, preparation of 198
- Lindo-Gladding method for potash 223
- Liquors, fermented 176
- Lupton, Mr. N. T., statement of, in regard to the feeding of cotton-seed meal 148

M.

- Magnesia mixture, preparation of 225
  - nitrate of 226
- Malic acid, determination of 216
- Mansfeld's method for the examination of butters 138
- Members, list of 5
- Milk analysis, analytical methods for 158
  - apparatus for 156
  - methods for 212
- Milk, analysis of, with a view to determining adulterations 167, 168
  - behavior of the sales of, with caseine 151
  - buffalo's, composition of 152
  - citric acid in 155
  - composition of, on the Ontario Experiment Farm 153
  - condensed, composition of 149
  - cow's, the limits of specific gravity of 154
  - detection of bicarbonate of soda in 170
  - determination of fat in, by Parson's method 164–166
  - estimating caseine in, by Kjeldahl method 170
  - estimation of albuminoids in 169
    - the fat in 162, 163
      - by the gravimetric method 159
        - Rose's method 160, 161
        - Short's method 166
        - Soxhelet's areometric method 163
        - Soxhelet, Engström and Schmœger-Neubert methods 158
        - Werner-Schmid method 158
  - fat in 154
  - fibrine in 155
  - from Constadt, report on 153
  - mean composition of 153
  - nature of 154

Page.

Milk, portion of dairy report relating to ... 149
quantitative determination of the addition of water to, and of the removal of cream therefrom ... 168
solids in ... 152
sugar, assimilation of ... 152
supply, of Paris ... 152
Moisture in cattle foods, remarks on ... 26
fertilizers, method for ... 224
Molybdic solution, preparation of ... 225
Myers, Mr. John A., address of ... 8
remarks of ... 185

N

National chemical society, discussion of the report of the committee on the organization of ... 68, 69
report of the committee on the organization of ... 65
views of Mr. F. W. Clarke, in regard to ... 66–68
Nicholson, Mr. H. H., letter from, in regard to butter analyses ... 118
Nitrates, determination of ... 87
Nitric acid, use of, in destroying organic matter ... 50
Nitrogen, absolute, method for ... 88
or cupric oxide, method for ... 217
albuminoid ... 226
discussion of report on ... 94, 95
Kjeldahl method for ... 218–221
modified to include nitrogen of nitrates ... 220, 221
methods for the determination of ... 217
notices of analytical methods for the determination of ... 85–88
remarks on analytical results obtained by various methods for the determination of ... 90–93
Ruffle method for ... 221
soda-lime method for ... 88, 221–223
tables, showing the results obtained in the samples furnished by Mr. M. A. Scovell for the determination of ... 89, 90
Nitrogenous matter in milk, estimation of total ... 212

O

O'Brine, Mr. David, letter from, in regard to butter analyses ... 118
Officers and reporters of the Association of Official Agricultural Chemists ... 189
election of ... 185
Official methods of analysis ... 193
Oleomargarine, method for the analysis of ... 99
Order of business, committee on ... 14
Organic matter, discussion of various methods for destruction of, in fetilizer analysis ... 50, 51

P.

Petroleum ether, temperature of distillation of ... 41, 42
use of ... 33, 40
spirit, use of ... 39, 40
Phosphoric acid, determination of citrate insoluble ... 224, 225
soluble ... 225
total ... 225
water soluble ... 224

Page.

Phosphoric acid, determinations, oxidation of organic matter in 98
    discussion of methods for the determination of water soluble. 58–63
        report on 49–57
    method for 224
    report on 44–49
    sieve for preparation of samples for determination of 224
Polariscope, selection of 193
Polarization, double 195
    of wines 216
Potash, alternate method for 223, 224
    in muriates, determination of 223
        superphosphates, determination of 223
        the sulphate, kainite, etc., determination of 223
    method of Lindo as modified by Gladding 226
    recommendations of the reporter on 83
    statement of the reporter on 79–83
    tables showing the results obtained in samples furnished by Mr. Jenkins for the determination of 81, 82
Prefatory note 3
Programme of order of business 15
Protein in cattle foods, remarks on 29

R.

Refractive index 211
Richards, Mr. Edgar, on the withdrawal of alcohol from bond 71
Rising, Mr. W. B., report of, on fermented liquors 173–176
Ruffle method, for nitrogen 221

S.

Samples, discussion of preparation of, in phosphoric acid determinations 52–57
    preparation of, for analysis 52
Sampling fertilizers, discussion of, by Mr. Frear 52
    in sugar analysis 199
Scovell, Mr. M. A., letter from, in regard to butter analyses 131, 132
    recommendations of, for the substitution of pumice stone for granulated zinc in the determination of nitrogen (Kjeldahl method) 95
    report of, on nitrogen 85–93
Schweitzer, Mr. Paul, letter from, in regard to butter analyses 122–124
Sieve adopted for use in the preparation of samples 57
Soda, caustic, where obtainable 98
Sorghum seed, means of 72 analyses of 83
Specific gravity (sugar analysis) 196
    of wines 215
Squibb's ether, use of 34
Starch, estimation of, in cattle foods 20
Succinic acid, determination of 216
Sucrose, determination of, by inversion 197
Sugar analysis, adjustment of instruments for 193, 194
    apparatus for 193
    discussion of paper of Mr. Ross on 183, 184
    methods for 193
    notes on 179
    re-agents for 193

Page.
Sugar, determination of, in wines ... 216
  estimation of, in cattle foods ... 20, 21
    milk ... 214

T.

Tannin, determination of, in wines ... 216
Tartaric acid, determination of ... 216
Temperature recommended for drying substances ... 38, 39
Total solids (sugar analysis) ... 196

U.

Uniformity of methods, importance of ... 34

V.

Volatile acids, alternate method of determining the ... 207
  estimation of ... 204
  in the food of milch cows ... 141
  of wines, determination of ... 215
  variation of, in butter fat ... 111
Voorhees, Mr. E. B., remarks of, on nitrogen determinations (Kjeldahl method). 95, 96

W.

Water, estimation of, in cattle foods ... 21, 23
    milk ... 212
      alternate methods for ... 212, 213
  temperature at which determined ... 36, 37
Weighing glass, precautions necessary in ... 37
Wiley, Mr. H. W., appointment of, as secretary ... 30
  recommendations of, in regard to methods for milk analyses adopted ... 171, 173
  report of, on dairy products ... 98–173
Willits, Hon. Edwin, address of ... 6
Wine analysis, methods of ... 215
Wines, abnormal constituents of ... 217
  butyric acid in diseased ... 217
  chlorine in ... 216
  citric acid in ... 216
  coloring matter of ... 216
  determination of acidity of ... 215
    alcohol in ... 215
    extract of ... 215
    glycerine in ... 215
    specific gravity of ... 215
    tannin in ... 216
    volatile acids of ... 215
  general methods of determining the purity of ... 174
  gums in ... 216
  inorganic matter of ... 216
  lactic acid in ... 217
  lime, magnesia, and phosphoric acid in ... 216
  mannite in ... 217
  polarization of ... 216
  potash in ... 216
  sulphuric acid in ... 216